OIL ANALYSIS

AUGUSTUS H. GILL

Prof. A. Lawrence Lowell
with the compliments
of the author.

A SHORT HAND-BOOK OF OIL ANALYSIS

BY

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PREFACE TO THE THIRD EDITION.

THE book has been thoroughly revised and, by consultation with the literature, brought up to date. Some sixteen pages of new matter have been added, including the latest methods of carrying out the iodine test, the bromine number, Halphen's test for cotton-seed oil, and minor changes throughout the volume.

BOSTON, September, 1903.

PREFACE TO THE FIRST EDITION

This little book was written primarily to meet needs of the author's own classes. It is given the public in the belief that there is a demand fc concise manual for the analysis of oils, which sł give the methods of applying the usual physical a chemical tests to the mineral as well as to the anii and vegetable oils.

It is not designed to take the place of any of existing books, but rather to serve as an introd. tion to them, more especially to Benedikt-Lew: witsch, which is to the oil chemist what Freseı is to the analytical chemist, and to which the wı wishes to express his own indebtedness. '[writings of Schaedler, Redwood, Allen, and Bra have also been freely consulted. Only the n commonly occurring oils are discussed, and t as regards their preparation, properties, analy constants,—the highest, lowest, and average b given,—and finally their uses and adulterants.

In the use of this book it is assumed that the student is thoroughly familiar with the usual operations of volumetric and gravimetric analysis, and has attained some proficiency in organic chemistry.

Acknowledgments are due to Mrs. Ellen H. Richards for hints and suggestions, and to Mr. William L. Root for assistance in reading the proof.

BOSTON, November, 1897.

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A SHORT HAND-BOOK OF OIL ANALYSIS.

PRELIMINARY OBSERVATIONS.

SAMPLES of oil are almost always handled in the trade, and frequently brought for analysis, in a four-ounce "oil vial." The student will pour out a portion from the quart can, after thorough shaking, into such a vial. Before proceeding with the tests to be later described, it is well to make certain preliminary observations upon this sample.

The *turbidity* showing the presence of water or of oils which imperfectly mix, and the *sediment*, either stearin or dirt, are to be noted; the *color* and *fluorescence*, or "bloom,"—the latter indicating the presence of mineral oils,—are next observed; the color varies from "water white," through straw, lemon-yellow, wine-red, to opaque.

The *odor* and *taste* may reveal to experts much concerning the source of the oil under examination;

for example, the fish oils, especially when warmed, have an unmistakable odor, and the presence of whale oil in sperm is often detected by its "nutty" taste.

By inverting the bottle when partially filled, and noting the way in which the oil runs off from the bottom and the number of drops, an approximate idea of the viscosity may be obtained.

PART I.

PHYSICAL AND CHEMICAL TESTS.

CHAPTER I.

PETROLEUM PRODUCTS.

(a) Burning Oils.

THE tests to be made are, in the order of their importance, flash test, fire test, specific gravity, distillation test, determination of sulphur, acidity, sulphuric acid test, test for mineral salts, determination of water.

Flash Test or Point.[1]—By flash point we understand that temperature to which an oil must be heated to give off vapors which, when mixed with air, produce an explosive mixture. The results of this test will vary according to the quantity of air over the surface of the oil, and whether this be moving or still; also according to the distance of

[1] The flash point is oftentimes a valuable means of detecting the admixture of substances; for example, 0.1 per cent. ether in alcohol may be discovered by this test. See Table II.

the testing flame from the surface of the oil. Furthermore, the size of the flame, the length of its time of action, its form and dimensions, and, lastly, the manner of heating of the oil, will all influence the result.[1]

From the above statement and that of Dudley[2] the following points are to be especially noted :

1. *The Rate of Heating.*—The faster the oil is heated the lower will be the flash point, as more vapor is driven out.

2. *Size and Depth of Cup.*—From a large and shallow cup the liquid evaporates faster; hence the lower will be the flash point. The most constant results are obtained from a deep cup about half filled.

3. *Quantity of Oil.*—The larger the amount of oil the more vapor will be driven out; hence the lower will be the flash point.

4. *Distance of Testing Flame.*—The nearer or—what amounts to the same thing—the larger the testing flame the lower will be the flash point. A large flame may produce local superheating.

5. *Point of Application of Testing Flame.*—The flame should be applied at the edge, as the mixture of air and vapor is more complete; this is best

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[1] Engler and Haase, Fres. Zeit., xx. 8 (1881).

[2] American Engineer and Railroad Journal, lxiv. 180 (1890).

effected by drawing the flame diametrically across the top of the cup. Dr. Dudley cites an instance in which the flash point obtained was considerably too high, owing to the fact that the testing flame was first applied in the centre of the cup.

6. *The thermometers* used should be frequently compared with a standard instrument.

7. *Draughts* should be carefully avoided.

Barometric changes are for practical work, negligible, each five millimeters between seven hundred and forty-five and seven hundred and seventy-five millimeters, causes a variation of but 0.1° C.

Lenz[1] states that the initial temperature of the oil is of importance, and as a result of several hundred determinations recommends cooling the oil contained in the flashing cup to 0° C. before making the test. In case the oil contain water, it must be removed by treatment with calcium chloride or sulphate.

The apparatus in use in this country are divided into two classes,—covered testers, in which the cup is covered with a perforated metal or glass plate, and open testers, in which the cup is not so covered. In the author's opinion the covered testers are the more scientific and give the more concordant results, and should be made the standard instruments.

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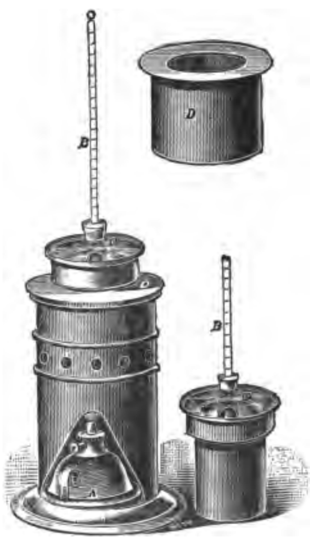
[1] Fres. Zeit., xxv. 265 (1886).

Covered Testers.—One of the best forms of testing apparatus is that devised by the Michigan State Board of Health in 1873, modified by Dr. A. H. Elliott, and now known as the "New York State Board of Health Tester," shown in Fig. 1.

FIG. 1.



New York State Board of Health tester.

Description. — It consists of a copper oil cup, *D*, holding about ten ounces, the quantity usually contained in lamps, heated in a water-bath by a small Bunsen flame. The cup is provided with a glass cover, *C*, carrying a thermometer, *B*, and a hole for the insertion of the testing flame,—a small gas flame one-quarter of an inch in length.

Manipulation. — After describing the apparatus minutely, the regulations of the New York State Board of Health say,[1] "(2) The test shall be applied according to the following directions:

[1] Report of the New York State Board of Health, 1882, p. 495.

"Remove the oil cup and fill the water-bath with cold water up to the mark on the inside. Replace the oil cup and pour in enough oil to fill it to within one-eighth of an inch of the flange joining the cup and the vapor-chamber above. Care must be taken that the oil does not flow over the flange. Remove all air-bubbles with a piece of dry paper. Place the glass cover on the oil cup, and so adjust the thermometer that its bulb shall be just covered by the oil.

"If an alcohol lamp be employed for heating the water-bath, the wick should be carefully trimmed and adjusted to a small flame. A small Bunsen burner may be used in place of the lamp. The rate of heating should be about two degrees per minute, and in no case exceed three degrees.

"As a flash torch, a small gas jet one-quarter of an inch in length, should be employed. When gas is not at hand employ a piece of waxed linen twine. The flame in this case, however, should be small.

"When the temperature of the oil has reached 85° F. the testings should commence. To this end insert the torch into the opening in the cover, passing it in at such an angle as to well clear the cover, and to a distance about half-way between the oil and the cover. The motion should be steady and uniform, rapid and without any pause. This

should be repeated at every two degrees' rise of the thermometer until the thermometer has reached 95°, when the lamp should be removed and the testings should be made for each degree of temperature until 100° is reached. After this the lamp may be replaced if necessary and the testings continued for each two degrees.

"The appearance of a slight bluish flame shows that the flashing point has been reached.

"In every case note the temperature of the oil before introducing the torch. The flame of the torch must not come in contact with the oil.

"The water-bath should be filled with cold water for each separate test, and the oil from a previous test carefully wiped from the oil cup."

Open Testers.—The Massachusetts statute is by no means as definite as that of New York; the courts have decided that custom fixes the method of testing. The law says,[1] "No person shall offer for sale . . . illuminating oils made from coal or petroleum which will evaporate a gas under 100° F. [that is, the flashing point is 100° F.—A. H. G.], or ignite at a temperature of less than 110° F., to be ascertained by the application of Tagliabue's or some other approved instrument."

[1] Revised Statutes of Massachusetts, 1902.

Manipulation.—Tagliabue's open tester (Fig. 2) is the official instrument. This is similar to the preceding, except that it is smaller, has no cover, and a glass oil cup.

FIG. 2.

Tagliabue's open tester.

The water-bath is filled as before, and the oil cup to within three-thirty-seconds of an inch of the top. The heating flame is adjusted so that it is three-fourths of an inch high, and the heating proceeded with at the rate of two and a half degrees per minute, until 97° F. is reached, when the test flame is applied and the testings made every three degrees until the flash point is reached. The whole time of making the test should be half an hour.

Fire Test.—The fire test of an oil is the temperature at which it will give off vapors which when ignited will burn *continuously*. It is made by continuing to heat the oil (the cover being removed in the case of a closed tester without slipping out the thermometer) at the same rate after the flash test is made and noting the point as indicated above. The flame is extinguished by a piece of asbestos board and the heating discon-

tinued. In the case of many illuminating oils this point is from 10° to 20° F. higher than the flash point.

Notes.—In the case of "Mineral Sperm" (300° F. fire test petroleum) these tests should be made with the instrument for lubricating oils (page 36). The heating should be at the rate of 15° F. per minute, and the testing flame first applied at 230° F., and then every seven degrees until the flashing point is reached.

The most satisfactory way of making these tests is to place the watch upon the desk and read the thermometer at the expiration of every minute, noting each reading down in the proper column in the laboratory note-book.

Specific Gravity.—This is usually effected by the hydrometer; a hydrometer jar is four-fifths filled with the oil, a Baumé hydrometer introduced into it, and the depth read off to which the instrument sinks in the oil. This may be effected by placing a strip of white paper back of the jar and noting the point at which the lower meniscus of the oil touches the scale. The temperature of the oil is taken at the same time, and in case it be not 60° F. (15.5° C.), for every increase of 10° F. (5.5° C.) subtract 1° Baumé from the hydrometer reading. The specific gravity may be found by the formula $\frac{140}{130 + B^\circ}$, B° representing the reading Baumé.

Notes.—Inaccurate graduation may cause an error of 0.001, but if the instrument be carefully calibrated it is accurate to 0.0002.[1] The student will make this test upon the oil at the ordinary temperature and correct the gravity for temperature as given above. In practice this can be done by Tagliabue's "Manual for Inspectors of Coal Oil," which gives the readings at 60° F. for any gravity from 20° to 100° Baumé, between 20° F. and 109° F.

Distillation Test.—As a means of evaluating samples of kerosene, Beilstein[2] recommends the fractional distillation of two hundred cubic centimeters, using a tower. As the method of Engler is more frequently employed, that will be described.

Apparatus.—Boiling flask, six and five-tenths centimeters in diameter, with neck fifteen centimeters long, and with the side tube about nine centimeters from the springing of the bulb; Liebig condenser; burette, or tall twenty-five cubic centimeter graduate; thermometer; small lamp with a shield.

Manipulation.—One hundred cubic centimeters of the oil are measured into the boiling flask and distilled at the rate of two to two and five-tenths cubic centimeters per minute, the distillate being caught in the burette or graduate. When the distil-

[1] Wright, Jour. Soc. Chem. Ind., xi. 302 (1892).

[2] Fres. Zeit., xxii. 309 (1883).

lation is to be broken, the lamp should be taken away and the temperature allowed to sink twenty degrees and again brought to the breaking or fractionating point, as long as any considerable quantity goes over. The distillation is first broken at 150° C., and then each fifty degrees until 290° C. is reached; in this way a much better idea of the value of the oil is obtained than if the distillation were allowed to proceed continuously between these points. The lighter portions, for example, those between 150° and 200°, burn much better than those between 250° and 290°; the heavy portions of American petroleum burn much better than those of the Russian oils.

The averages from four samples of Caucasian and ten samples of American oils subjected to this test were as follows, in per cent. by volume:[1]

	Below 150° C.	150°–290° C.	Above 290° C.
Caucasian petroleum	8.0	86.6	5.4
American petroleum	16.9	57.1	26.0

Determination of Sulphur.—In addition to the preceding tests, Professor Peckham[2] considers the determination of sulphur to be of considerable importance. The deleterious effects of the oxides of

[1] Veith, "Das Erdoel," p. 244.

[2] Report upon Petroleum.

sulphur upon hangings and bindings is well known, sulphuric acid being their ultimate product. The sulphur exists in combination, partly as compounds formed from the sulphuric acid used in refining[1] and partly as sulphides already formed in the oil. Its qualitative detection may be effected by heating the oil to its boiling point with a bright piece of sodium or potassium. If sulphur compounds be present, a yellowish layer is formed upon the metal. After cooling add distilled water drop by drop until the metal is dissolved, and test for sulphides with sodium nitro-prusside.

For the quantitative determination of sulphur many methods have been proposed. Engler[2] and Kissling[3] burn the oil in an apparatus similar to that used for the determination of sulphur in illuminating gas. Aufrecht[4] distils the oil with sodium bicarbonate, which takes up the sulphur.

Mabery[5] states that for oils containing more than 0.01 per cent. of sulphur the well-known method of Carius—the oxidation in a sealed tube with fuming

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[1] Vohl, Dingler Polyt. Jour., ccxvi. 47 (1875).

[2] Chem. Zeitung, xx. 197; abstr. Jour. Soc. Chem. Ind., xv. 883.

[3] Ibid., 199; abstr. Analyst, xxi. 162 (1896).

[4] Pharm. Zeitung, xli. 469; abstr. Jour. Soc. Chem. Ind., xv. 680 (1896).

[5] Am. Chem. Jour., xvi. 544 (1894).

nitric acid—leaves little to be desired. For oils containing a smaller percentage than this he employs a modification of Sauer's method—the combustion of the oil in a stream of air—and subsequent absorption of the products in standard sodium hydrate. The percentage of sulphur should not exceed 0.05; Engler, *loc. cit.*, found 0.02 to 0.03 in the Pennsylvania, and 0.04 to 0.05 in the Lima kerosenes.

Detection of Acidity.—Shake equal quantities of oil and warm water in a test-tube, pour off the oil, and test the water with litmus paper. If the water be strongly acid, the quantity may be determined as in "Free Acid," page 66.

The acid in this case is most probably sulphuric, coming from the refining process.

Sulphuric Acid Test.—The object of this test is to judge of the degree of refinement of the oil, a perfectly refined oil giving little or no color when submitted to the process. One hundred grams of oil and forty grams of sulphuric acid, 1.73 specific gravity, are shaken together for two minutes in a glass-stoppered bottle and the color of the acid noticed. In accurate work this color is matched by solutions of Bismarck brown.[1]

Mineral Salts.—Salts of calcium or magnesium when dissolved in the oil diminish its illuminating

[1] Jour. Soc. Chem. Ind., xv. 678 (1896).

power; their action is to form a crust on the wick and prevent access of air.

Redwood[1] states that 0.02 gram of either of these salts in one thousand grams of oil diminishes the illuminating power thirty to forty per cent. in eight hours.

They are determined by distilling one hundred or two hundred cubic centimeters of the oil down to about twenty cubic centimeters, evaporating and igniting this residue, and subsequently treating with hydrochloric acid. The calcium and magnesium are then determined in the usual way.

Determination of Water.[2]—Allen[3] states that water in oils may be determined by the addition of a weighed amount of gently ignited plaster of Paris. This is washed with a little gasolene, dried at a gentle heat and reweighed, the gain in weight being the water present.

REFERENCES.

In addition to the literature previously given, the student is referred to the following:

ELLIOTT, A. H., New York State Board of Health Report, 1882, pp. 449–496. This gives comparative tests of the various testers and a *résumé* of bibliography and patents up to that year.

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[1] Dingler Polyt. Jour., cclxv. 427 (1887).

[2] See, also, Davis, Jour. Am. Chem. Soc., xxiii. 487 (1901).

[3] Commercial Organic Analysis, ii. 491.

Peckham, S. F., "Report on the Production, Technology, and Uses of Petroleum and its Products," U. S. Census Report, 1885.

Thörner, W., Chemiker Zeitung, x. 528, 553, 573, 582, 601; abstracted in Jour. Soc. Chem. Ind., v. 371 (1886). "Petroleum as an Illuminating Agent."

Newbury and Cutter, Am. Chem. Jour., x. 356 (1888). "On the Safety of Commercial Kerosene Oil."

Aisinman, Chem. technische Vortäge, ii. 325. German methods of testing.

CHAPTER II.

PETROLEUM PRODUCTS.

(b) Lubricating Oils.

THE tests to be made are, in the order of their importance, viscosity, specific gravity, evaporation, cold test, flash test, fire test, test for soap, test for antifluorescents, friction test.

The office of a lubricant is to prevent the attrition of axle and journal by interposing itself between them in a thin layer, upon which the shaft revolves. The ideal lubricant is that which has the greatest adhesion to surfaces and the least cohesion among its own particles, or, as the practical man expresses it, the most fluid oil that will do the work and stay in place. The determination of its viscosity or "body" is then of the first importance.

Viscosity is the degree of fluidity of an oil or its internal friction. It is independent of the specific gravity of the oil, although this in the pipette instruments influences the time of efflux. Within certain limits it may be taken as a measure of the value of oil as a lubricant, by comparing the viscosity of the oil under examination with that of other oils

which have been found to yield good results in practice.

The instruments employed for its determination may be divided into two classes,—pipette viscosimeters, giving the time of efflux, as those of Saybolt, Engler, and others, and torsion viscosimeters, giving the retardation due to the oil, those of Napier and Doolittle.

Of these but two, the Saybolt and Doolittle, will be minutely described.

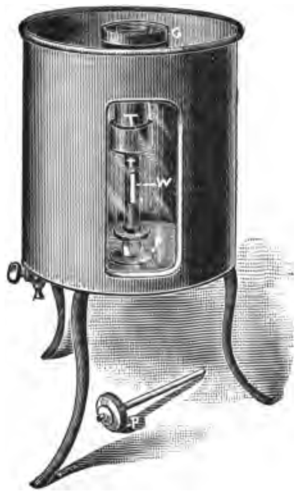
The Saybolt Viscosimeter.[1]—This is made in three forms, A, B, and C. Apparatus "A" is the standard for testing at 70° F. Atlantic Red, Paraffine, and other distilled oils. "B" for testing at 70° F. Black Oils of 0°, 15°, 25°, and 30°, Cold Test, and other reduced oils up to, but not including, Summer Cold Test Oil. Apparatus "C" is used for testing at 212° F. Reduced, Summer, Cylinder, Filtered Cylinder, XXX Valve, 26.5° Bé., and other heavy oils.

Apparatus "A."—*Description.*—The "A" apparatus (Fig. 3) consists of a brass tube, *T*, containing about sixty-six cubic centimeters, and about three centimeters in diameter and eight centimeters long, forming the body of the pipette. It is connected at

[1] Redwood, Jour. Soc. Chem. Ind., v. 124 (1886).

the bottom with a smaller tube, *t*, having a window, *w*. This pipette is screwed into the piece *p* carrying the jet, 1.75 millimeters in diameter; the lower part of this piece is expanded at the bottom to admit of the insertion of a cork. The upper part of the pipette is perforated with a number of small holes leading to a gallery, *G*, five centimeters in diameter and one and three-tenths centimeters deep. This enables a workman to fill the apparatus to the same point every time. This pipette is held by *p* in a tank of water eighteen centimeters high and twenty centimeters in diameter, also provided with windows to observe the efflux of the oil.

Fig. 3.



Saybolt's "A" viscosimeter.

A tin cup with spout, thermometer, pipette with rubber bulb, stop-watch, and beaker for waste oil, complete the outfit.

Manipulation.—Having the bath of water prepared at 70° and the oil in the tin cup about 69.5°, clean the tube out with some of the oil to be tested by

using the plunger sent with the instrument. Place the cork air-tight in the lower outlet tube and pour the oil into the tube proper until it flows into the overflow cup.

By stirring with the thermometer bring the oil to exactly 70°, remove the thermometer, and draw with a pipette the surplus oil in the overflow cup down below the overflow holes. The temperature still remaining constant, with the watch in the left hand, draw the cork with the right and simultaneously start the watch. Towards the end of the run, watch the peep-hole closely through the window in the bath, and at the first appearance of space not filled with oil in the glass outlet tube stop the watch.

Apparatus "C."—Standard for testing at 212° F.

Description.—This is very similar to the preceding apparatus. The top is closed around the oil gallery, the windows omitted, and a steam connection provided. The quantity of oil is determined by allowing it to run into a sixty cubic centimeter graduated flask. The size of the jet is three-sixty-fourths of an inch.[1]

Manipulation.—Fill the bath with water and attach steam inlet to bottom cock. The upper outlet is for exhaust and overflow.

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[1] Stillman, Jour. Anal. and App. Chem., v. 322 (1891).

With the water boiling and the bath thermometer registering 212°, and the oil to be tested having been put through the strainer into one of the tin cups, pour some of the oil into the tube and clean out with the plunger sent with the instrument; never use other than the plunger in cleaning.

Place the cork air-tight in the lower outlet tube, and pour the oil into the tube until it overflows into the overflow cup. Allow the oil to heat until the temperature is 210°, stirring the oil with the thermometer during the heating. Having the bath at 212° and the oil not below 210°, remove the thermometer. Draw the surplus oil from the overflow cup with a pipette down to and below the overflow holes; this insures a positive starting-point.

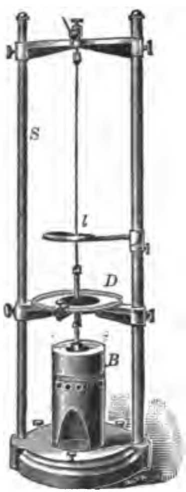
Place the sixty cubic centimeter flask under and directly in a line with the outlet jet, and as close to the jet as it is practicable, to permit of room for drawing the cork. With the watch in the left hand, draw the cork with the right and simultaneously start the watch. The time required for the delivery of sixty cubic centimeters is the viscosity. The tube should be cleaned out before each test with some of the oil to be tested.

Notes.—Instead of timing the oil as given in the directions above, the writer has found it better to start the watch, and the instant the second hand

crosses the sixty seconds mark *twist* out the cork with the right hand.

The tube should be cleaned out before each test with some of the oil to be tested, using the plunger *P* for this purpose. Black oils or any oil containing sediment should be carefully strained before testing or "running," as it is technically termed. The instruments should be carefully guarded from dust when not in use.

FIG. 4.



Doolittle's torsion viscosimeter.

Doolittle's Torsion Viscosimeter.[1]—*Description.*—The apparatus consists of a cylinder (Fig. 4) rotating in the oil, and a graduated disk, *D*, to measure the amplitude of rotation. These are supported by a fine piano wire from the substantial stand *S*, provided with levelling screws; a lens, *l*, enables the graduations on the disk to be read more accurately, and a bath, *B*, filled with water or oil serves to maintain any desired temperature. The instrument should be so adjusted that it will read within one-half degree of the zero point on either side of it when vibrating through an arc of one hundred

[1] Doolittle, Jour. Am. Chem. Soc., xv. 173, 454 (1893).

and eighty degrees; this can be effected by loosening the set screw at the top and turning the pin which holds the wire.

Manipulation.—Immerse the friction cylinder in the oil by slipping its stem into the stem of the disk, and adjust the temperature very carefully to the point at which it is desired to determine the viscosity; great care must be taken to keep this temperature constant during the test. Either a water-bath or bath of lard oil—according to the temperature desired—may be used. The oil in the cup should cover the cylinder with a layer three-sixteenths of an inch deep when it is swinging freely, and it should be in the centre of the cup.

By lifting the milled head at the top of the instrument out of the notch, and turning it completely around from right to left until it drops into the notch again, the wire is rotated three hundred and sixty degrees. By raising the disk by means of the cam the friction cylinder will rotate in the oil by virtue of the torsion of the wire. The disk will rotate three hundred and sixty degrees and a portion of another arc, which latter is the first reading, —the end of the first swing $= 355.6°$ right. The left-hand swing is ignored, and the arc on the next swing to the right $= 338.2°$ right is read. The retardation produced by the oil is $355.6° - 338.2° = 17.4°$. The

vibrations should now be stopped, and the head should be turned in the opposite direction and the readings to the left taken, and the average of the two considered as the retardation of the oil.

The results are expressed in the number of grams of sugar contained in one hundred cubic centimeters of sugar syrup at 60° F., its viscosity being taken at a temperature of 80° F. In the example cited, 17.4° (with the small cylinder) represents a viscosity of 65.6; this means that if 65.6 grams of granulated sugar were dissolved in water at 60° F., made up to one hundred cubic centimeters, and then heated to 80° F., its viscosity would be the same as that of the oil under examination.

The readings of the first and second swings are to be taken, as later vibrations give different results. The wire and cylinder should be handled with great care, as they are very sensitive to abuse. The wire should be greased with tallow occasionally, and in case of a new instrument, restandardized after six months' use. In case a new wire is inserted the instrument must be recalibrated. When not in use the 0° point should be kept under the index, the disk upon its supports, and the wire without torsion.

Traube's Viscosimeter.—Dr. T. Traube,[1] of

[1] Traube, Zeit. d. Ver. deutsch. Ing., xxxi. 251; abstr. Jour. Soc. Chem. Ind., vi. 414 (1887).

Hanover, uses a pipette viscosimeter consisting of a vertical bulb with a long horizontal capillary jet, it being contained in a trough to keep the temperature constant. About eight cubic centimeters of oil are used for a test, and forced through the jet under a pressure of sixty centimeters of water. This jet is thirty centimeters long and of various diameters, there being three pipettes with jets 1.5, 0.8, and 0.5 millimeters in diameter, according to the kind of oil to be tested.

Wright[1] states that the results correspond more closely to those obtained on a friction machine than those of any other instrument,—a statement which the author's experiments would seem to confirm. The instrument certainly is more sensitive than any with which the author is acquainted.

Specific Gravity.—1. *By the Hydrometer.* See page 16.

2. *By the Westphal Balance.*—This is a specially constructed instrument with a glass plummet carrying a thermometer counterbalanced by a weight. Upon immersing the plummet in a liquid the positions of weights, which must be added to restore the equilibrium, represent the specific gravity di-

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[1] Oils, Fats, and Waxes, p. 109.

For the absolute viscosimeter, see "Lubrication and Lubricants," Archbutt and Deeley, pp. 127–137.

rectly. The largest weight represents the first decimal place, the next the second, and so on. The instrument is placed upon a level table, and by means of the levelling screw is brought into adjustment,—*i.e.*, so that the point upon the beam is exactly opposite the point upon the fixed part.

The plummet is now placed in the vial or balance jar containing the oil, cooled to 15.5° C., hung upon the balance, being careful to completely immerse it in the oil, weights added to restore the equilibrium, and the specific gravity read off as above described.

Notes.—In using the instrument care should be taken to place the riders at right angles to the beam, otherwise an error of 0.0005 may be introduced; furthermore, the loop upon the knife-edge should always be in the same position. In buying an instrument the spaces upon the beam should be tested with dividers to insure their equality, otherwise serious errors may be caused.[1] The limit of accuracy is about 0.0005.[2]

Care should also be taken that the plummet does not touch the sides of the jar or vial.

For solid fats and some oils the specific gravity is taken at 100° C., using a special plummet.

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[1] Allen, Analyst, xiv. 11; Stock, ibid., 50 (1889).

[2] Richmond, ibid., 65.

McGill[1] states that the balance is more sensitive for viscous oils when the specific gravity of the plummet is 4.0 than when it is 2.0.

Evaporation Test.[2]—The object of this test is to determine what percentage of an oil—more especially a spindle oil—is volatile when exposed to nearly the same conditions as it is on a bearing.

Apparatus.—Flat watch-glass; annular disks of filter-paper, one and five-eighths inches outside diameter, with inside hole five-eighths of an inch in diameter, which have been standing in a sulphuric acid desiccator for several days; and an oven with thermometer and thermo regulator.

Manipulation.—The watch-glass and paper are weighed,—to tenths of a milligram,—and about 0.2 gram of oil brought upon it by dropping from a rod, and *accurately* weighed. The watch-glass is now placed in an air-bath, the temperature of which remains nearly constant at 60° to 65° C. (140° to 150° F.), and heated for eight hours. It is then cooled and reweighed, the loss being figured in per cent. No oil should be passed which gives an evaporation of more than four per cent.

The following table of results upon some spindle oils shows the relation of gravity, flash point, and evaporation:

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[1] Analyst, xxi. 156 (1896).

[2] See, also, Archbutt, Jour. Soc. Chem. Ind., xv. 326 (1896).

Gravity.	Flash, ° F.	Evaporation.
. .	298	7.0 per cent.
.846	318	4.4 "
. .	348	2.0 "
.852	348	1.0 "
.856	336	1.4 "
.862	352	0.9 "
.866	366	1.7 "
.870	384	0.8 "
.882	364	1.7 "

Notes.—The temperature employed, 65° C., is approximately that attained by a bearing (in a spinning frame) after running two hours, thus leaving the oil exposed to it for eight hours, assuming a ten-hour day.

The test is important to the insurance underwriter, because it measures the amount of inflammable material sent into the air, and hence the liability to cause or aid conflagrations; it is important to the mill-owner, as it indicates the quantity of oil left upon the bearing, hence serving its purpose.

Cold Test.—This may be defined as the temperature at which the oil will just flow.

Apparatus.—Four-ounce vial; thermometer; battery jar; freezing mixture.

Manipulation.—The four-ounce vial is one-fourth filled with the oil to be examined, the short, rather heavy, thermometer inserted in it, and the whole

placed in a freezing mixture. When the oil has become solid throughout, the vial is removed, the oil allowed to soften, and thoroughly stirred until it will run from one end of the bottle to the other. The reading of the thermometer is now taken by withdrawing it and wiping off the oil with waste to render the mercury visible.[1]

The *chilling point* is the temperature at which flakes or scales begin to form in the liquid, and is determined similarly, by cooling the liquid five degrees at a time.

Freezing Mixtures.—For temperatures above 35° F. use cracked ice and water; between 35° and 0° F. use two parts of ice and one part of salt; and from 0° to —30° F. use three parts of crystallized calcium chloride and two parts of fine ice or snow. A still more convenient means is by the use of solid carbonic acid dissolved in ether, giving —50° F. readily.

The preceding method is open to quite an error from the personal equation of each observer. To obviate this Martens[2] proceeds as follows:

The oil is poured into a U-tube one centimeter in diameter, sixteen centimeters high, with three

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[1] Dudley and Pease, Am. Eng. and R. R. Jour., lxix. 382 (1895).

[2] Mitt. d. k. tech. Versuchstation; abstr. Jour. Soc. Chem. Ind., ix. 772 (1890).

centimeters between the bends, to a depth of three centimeters; it is then placed in a freezing mixture, cooled, and connected with a blast at a constant pressure of three centimeters. The temperature at which the oil begins to flow under these conditions is considered as the cold test.

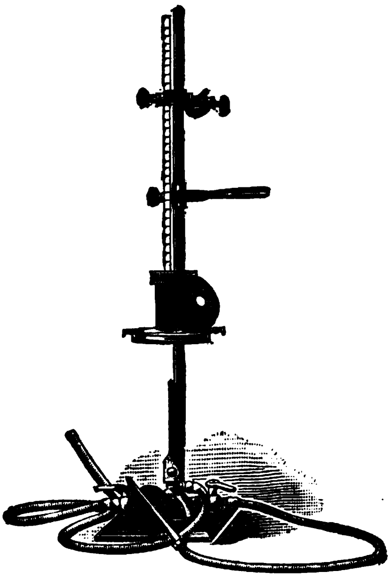
Flash Point.—Several forms of apparatus for testing the flash point of lubricating oils have been devised: Pensky-Martens's closed tester employing a stirrer is used in Germany. Martens states in a later article that stirring is unnecessary. Dudley and Pease use an open porcelain dish heated with a Bunsen burner.

Description.—The apparatus in use in the author's laboratory is similar to the New York State tester, and consists of a covered copper cup—shown at about one-tenth the size in Fig. 5—supported by gauze upon an iron stand and heated by a Tirrill burner.

Manipulation.—The cup is filled with oil to within three-eighths of an inch of the flange (in case of cylinder or oils flashing above 500° one-half inch), all air-bubbles removed, the flange and top of cup carefully wiped free of oil, the cover put on, and the thermometer inserted so that its bulb is half-way between the surface of the oil and bottom of the cup. The lamp is placed underneath, carrying a flame

about an inch in height, the bottom of the cup being two and a half inches from the mouth of the burner, and the heating commenced. The rate of heating should be 15° F. per minute, and may be readily

FIG. 5.



Flash apparatus.

regulated by the burner used. The testing flame should be first applied at 250° F., and then every half-minute until the flash point is reached. This is indicated by a slight puff of flame out of the testing hole.

Fire Test.—The cover is supported above the cup, and the heating and application of the testing flame continued as in making the flash test.

The method of recording is the same as in the case of the illuminating oils, one column for times and another for temperatures; the student is recommended to read again pages 9–16. Holde[1] finds that with oils flashing between 172° C. and 241° C. the exact quantity of oil used is of little importance. In these particular cases a difference of filling of thirteen cubic centimeters altered the flash point only 1–1.5° C.

It is worthy of notice that the free acid contained in an oil lowers its flash point apparently in proportion to the quantity present.

Detection of Soap.—To increase the viscosity of an oil,[2] resort is had to the use of "oil pulp," "oil-thickener," or "white gelatin," usually an oleate of aluminium, though other bases may be present. Its disadvantages are that it causes the oil to chill more easily and to emulsify, thus increasing the friction. Furthermore, it is precipitated by contact with water or steam, causing clogging of the machinery.

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[1] Jour. Soc. Chem. Ind., xvi. 822 (1897).

[2] In a case which came to the writer's notice the oil would not flow out of the Saybolt "A" apparatus at 70°, at 85° required 1167″, and at 110°, 181″.

The test depends upon the fact that the metaphosphates of the earthy and alkali metals and aluminium are insoluble in absolute alcohol.[1]

Reagent.—Saturated solution of metaphosphoric acid in absolute alcohol.

The test is applied as follows: Five to ten cubic centimeters of the oil to be tested are dissolved in about five cubic centimeters of 86° gasolene or ether, and about fifteen drops of the phosphoric acid solution added, shaken and allowed to stand; the formation of a flocculent precipitate indicates the presence of soap. An idea of the kind of soap can be often gained by adding an alcoholic solution of $PtCl_4$. If the precipitate becomes crystalline it is a potash soap; if it dissolves, soda, lime, or magnesia; if unchanged, alumina or iron.

For the accurate determination of these compounds a known weight of the oil must be ignited, the residue determined and quantitatively examined.

Caoutchouc.—Holde[2] states that one to two per cent. of unvulcanized caoutchouc is sometimes added to oils to increase their viscosity. This may be detected by adding three parts of alcohol to

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[1] Schweitzer and Lungwitz, Jour. Soc. Chem. Ind., xiii. 1178 (1894).

[2] Die Schmiermittel, p. 122.

four parts of the ethereal solution, whereby the rubber material is precipitated and may be dried and weighed.

Tests for Antifluorescents.[1]—*Reagent.*—Ten per cent. solution of potassium hydrate in alcohol.

It is often desired to remove the fluorescence or "bloom" from petroleum oils. This may be effected by refining with chromic acid, or more easily by the addition of a small quantity of nitro-naphthalene or nitro-benzene. The latter may often be detected by the odor.

The test is made by boiling about one cubic centimeter of the oil with three cubic centimeters of the alcoholic potash for one to two minutes. If either of the nitro compounds be present, a blood or violet-red coloration is produced: a pure mineral oil is changed only to yellow or brownish-yellow by this treatment. In case the characteristic color does not appear, the test should be repeated with larger quantities.

To ascertain if an oil be fluorescent, place a few drops upon a piece of hard rubber or other black surface and observe if any trace of blue color be perceptible.

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[1] Holde, Jour. Soc. Chem. Ind., xiii. 906 (1893).

Test for Fatty Oils.—To detect small quantities of fatty oil (one-quarter to two per cent.) Lux[1] recommends heating a few cubic centimeters of the oil for fifteen minutes with some bits of sodium in a test-tube in an oil-bath; a similar test is made with sodium hydrate. The temperature employed should be for light oils about 230°, for dark oils 250°.[2] In case fatty oil be present, the contents of one or both of the tubes solidify to a jelly of greater or less consistence according to the amount of fatty oil present.

Gumming Test.[3]—This is designed to give an idea of the amount of change that may be expected in a mineral oil when in use. These resinified products increase the friction of the revolving or rubbing surfaces.[4] It is applied after the manner of the Elaidin Test, by thoroughly mixing together five grams of the oil in a cordial glass with eleven grams of nitrosulphuric acid and cooling by immersion in a pan of water at 10°–15°. Brownish spots or, in case of a bad oil, masses, form around the edges and become red in the course of two hours. As shown by long practical experience, the oil

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[1] Zeit. f. Anal. Chem., xxiv. 357.

[2] Holde, Die Schmiermittel, p. 109.

[3] Gill, Jour. Am. Chem. Soc., xxiv. 467 (1902).

[4] Aisinman, Jour. Soc. Chem. Ind., xiv. 282 (1895).

showing the least tar is the best oil; it also absorbs the least oxygen.

Friction Tests.—The writer is inclined to doubt if friction tests are worth the outlay for a machine and the time expended in their execution. Without question they do determine the relative efficiency as regards lubricating power of different oils, but the conditions under which the test is made seldom occur in practice; the bearings upon which the oil is tested are as nearly perfect as can be made, and the feed and load are as regular as is possible; in other words, the conditions are ideal.

The lubricating power of an oil is so closely related to its viscosity[1] that the author believes that results of more practical value can be obtained by the determination of the viscosity of the oils, and subsequent observation of their behavior in actual use, than by the longer and more troublesome friction test.

In case, however, it be desired to make the friction test, the following machines, it is believed, will be found to be most satisfactory for the purpose.

For spindle oils and light lubricating oils, the Ordway-Woodbury machine,[2] described in the "Pro-

[1] Brannt, "Petroleum and its Products," p. 510; Woodbury, *vide infra.*

[2] Made by the Pratt & Whitney Co., Hartford, Conn.

ceedings of the American Society of Mechanical Engineers," 1880, 74, and 1884, 136; also in Brannt, "Petroleum and its Products," p. 480.

For heavy oils and railroad work, the large machine of Thurston,[1] described in his "Friction and Lost Work in Machinery and Millwork," p. 254; also in Brannt, p. 486; also in Archbutt and Deeley.[2]

For machines using a flooded bearing the Beauchamp-Tower machine, described in the "Proceedings of the Institution of Mechanical Engineers of Great Britain," 1883, 632; 1884, 29; 1885, 58; 1888, 173; 1891, 131; also in Archbutt and Deeley.[3]

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[1] Made by the Pratt & Whitney Co., Hartford, Conn.

[2] "Lubrication and Lubricants," 1901, pp. 322–330.

[3] Ibid., p. 333.

CHAPTER III.

ANIMAL AND VEGETABLE OILS.

THE tests most commonly employed for the identification of these oils are as follows: specific gravity, Valenta test, elaidin test, Maumené test, heat of bromination test, iodine number and saponification value.

In addition certain special and commercial tests are applied, as Bechi test, Baudouin test, free acid, spontaneous combustion and drying test.

Specific Gravity.—This is usually determined either by the Westphal balance (page 31) or by the *picnometer*.

A two-necked flask of fifty cubic centimeters capacity, having a thermometer carefully ground into one neck, the second one being a narrow tube bearing the mark, is most suitable. This is filled with the oil to be examined, cooled to 15.5° C.,[1] the excess of oil removed and weighed. If the weighings be made to 0.5 milligram and a correction applied for the expansion of the glass by the difference in temperature, = 15.5° — 4° = 11.5° = —0.025 per

[1] Allen (Organic Analysis, 33) states that a correction of 0.00064 can be made for each variation of 1° C.

cent. of the value obtained, the determination is accurate to 0.00002.[1]

For the determination of the specific gravity of small quantities of oil, the aræopicnometer of Eichhorn[2] may be used. This consists of an ordinary hydrometer having a stoppered ten cubic centimeter bulb between the empty and weighted bulbs. The stoppered bulb is completely filled with the oil at 15°, wiped dry, and the instrument placed in a hydrometer jar filled with water at 15° C., and the depth to which it sinks shows the specific gravity of the oil.

Valenta Test.[3]—Although considered by some to be unreliable, yet as the indication given by this test may be of value, it is worth the trouble of execution. It depends upon the solubility of the oil in glacial acetic acid.

Enough oil is poured into a test-tube to fill it to the depth of about one inch, the exact height being marked by the thumb; an equal quantity of glacial acetic acid is poured in, that is, until the acid reaches the point indicated by the thumb. A light thermometer is placed in the tube, and it is

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[1] Wright, Jour. Soc. Chem. Ind., xi. 300 (1892).

[2] Fres. Zeit., xxx. 216, abstr. (1891).

[3] Valenta, Dingler Polyt. Jour., ccliii. 418; also Jour. Soc. Chem. Ind., iii. 643 (1884).

heated until the oil dissolves,—shown by the liquid becoming homogeneous. The tube is now allowed to cool, and the point noted at which it begins to be turbid.

Castor oil is soluble at ordinary temperatures, while rape-seed and other cruciferous oils are insoluble even at the boiling point of the acid. The temperatures at which other oils become turbid are given in Table VIII.

Elaidin Test.—Although this is not a quantitative test, yet its ease of application and the conclusions which may be drawn from it render it valuable. It depends upon the change of the liquid olein into its solid isomer elaidin, and is especially applicable to olive and lard oils.

Poutet's Method.—Apparatus required.—Cordial glasses; short glass rods; scales; ten-inch agate-ware pan.

Manipulation.—Five grams of the oil are weighed —within two drops—into the cordial glass, seven grams of nitric acid, specific gravity 1.34, are then weighed into it, and two pieces of copper wire (0.6 to 1.0 gram) added. Place the glass in the pan of cold water at about 15° C., and stir about twenty to thirty turns, not only with a rotary movement but also with an up-and-down motion, so as to mix the oil and the evolved gas thoroughly. When the wire

has dissolved, add a second piece and stir as before. This second addition should furnish gas enough if the liquid has been kept cool and the stirring has been thorough.

At the end of the first hour pure lard oil will usually show flakes of a wax-like appearance, and upon standing without disturbance and at the same temperature for another hour, the oil will have changed to a solid white cake hard enough to bear several ounces weight or admit of lifting the glass and contents by the glass rod.

Most of the fish and seed oils yield a pasty or buttery mass separating from a fluid portion, whereas olive, almond, peanut, lard, sperm, and sometimes neat's-foot oil, yield a solid cake.

Instead of using nitric acid and copper, sulphuric acid of 46° Baumé, containing a little nitric acid and saturated at 0° C. with nitric oxide, may be employed.

A test should always be made at the same time with an oil of undoubted purity.

Notes.—If the oil be stirred too much or too frequently, it has no opportunity to form a hard cake.

Hübl states that all attempts to make the test a quantitative one have resulted in failure.

Mercury can be used instead of copper.

Cailletet's method,[1] in which a smaller quantity of oil is used and sulphuric and nitric acids allowed to act upon it in a boiling water-bath, cannot, in the experience of the writer, be depended upon to give reliable results.

Maumené Test.—While this, like the preceding, is not a quantitative test, yet the indications afforded by it are of more value in many cases than those obtained by quantitative methods, as, for example, the saponification value. It depends upon the heat developed by the mixing of the oil with strong sulphuric acid.

Apparatus required.—Small beaker seven and one-half to nine centimeters deep and of one hundred and fifty cubic centimeters capacity packed in an agate-ware cup with *dry* felt or cotton-waste packing; light thermometer; ten cubic centimeter graduate; scales.

Manipulation.—Fifty grams of the oil are weighed into the beaker to within two drops, and its temperature noted by the thermometer. Ten cubic centimeters of sulphuric acid are now run gradually into the oil,—allowing the graduate to drain five seconds,—the mixture being stirred at the same time, and the stirring continued until no further increase in temperature is noted. The highest point at which the thermometer remains

[1] Milliau, Jour. Am. Chem. Soc., xv. 156 (1893).

constant for any appreciable time is observed, and the difference between this and the initial temperature is the "rise of temperature." This varies with the strength of the acid employed, and to secure uniformity the results should be expressed by dividing the rise of temperature with the oil by the rise of temperature with water, and multiplying by one hundred. This is called the "specific temperature reaction." The rise of temperature with water is determined in the same manner as with oil, using the same vessel.

Notes.—In performing this test it is important that the oil and acid be of the same temperature, attained by keeping them beside each other in the same room.

The strength of acid should be as far as possible the same; it should be determined not by specific gravity, but by titration, as one hundred per cent. and ninety-four and three-tenths per cent. acid have the same specific gravity.

For concordant results the conditions should be the same, and the same apparatus should be used. In case the test is to be applied to a drying oil, it should be diluted one-half with a mineral oil, 25° paraffine, for example, thoroughly mixing them. The "rise of temperature" is then, the rise of temperature of mixture minus half the rise of tempera-

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ture of fifty grams of mineral oil, multiplied by two.

It is advisable to make a test at the same time with an oil of known purity. The student will perform this test upon the assigned oil in duplicate, reporting both the rise of temperature and "the specific temperature reaction." Results should agree within two or three per cent.

Sherman, Danziger, and Kohnstamm[1] have studied this method with the idea of eliminating the errors. Rather than dilute the oil with a mineral oil they dilute the acid, using one of eighty-nine per cent. The results obtained are a little lower for vegetable oils and a little higher for animal oils than those usually found with the strong acid as employed by Thomson and Ballantyne. Mitchell[2] uses an inert diluent—carbon tetrachloride—in a vacuum-jacketed tube and one-fifth the quantities; all oils are diluted. He finds that the results obtained are in close agreement with the bromine thermal values; further, that the test may be of use in determining the degree of oxidation of fats and oils, the figures becoming greater with the age of the oil.

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[1] Jour. Am. Chem. Soc., xxiv. 266 (1902).

[2] Analyst, xxvi. 169 (1901).

Data upon various oils will be found in Table VIII.

REFERENCES.

MAUMENÉ, Compt.-Rend., xxxv. 572 (1852).

ELLIS, Jour. Soc. Chem. Ind., v. 361 (1886).

THOMSON and BALLANTYNE, Jour. Soc. Chem. Ind., x. 234 (1891).

RICHMOND, Analyst, xx. 58 (1895).

MUNROE, Am. Pub. Health Ass'n, x. 236 (1884).

Heat of Bromination Test.—This test, which was proposed by Hehner and Mitchell,[1] consists in observing the rise of temperature when bromine is added to a solution of the oil in chloroform. It occupies a middle position between the Maumené, being more accurate than it, and the Hübl, than which it is less delicate; by multiplying by a factor, different for each instrument, the results obtained can be expressed in figures, which are a close approximation to those obtained by the Hübl method.

The process has not found extensive application, and for a description of the method of execution reference may be had to articles by Wiley, Jour. Am. Chem. Soc., xviii. 378 (1896), and Gill and Hatch, ibid., xxi. 27 (1899).

Iodine Number or Value.—This is the percentage of iodine absorbed by an oil; the method de-

[1] Analyst, xx. 146 (1895).

pends upon the fact that different oils absorb different amounts of the halogens; the process is mainly one of addition, although small quantities of substitution products are formed. For example, the unsaturated body olein, $(C_{17}H_{33}COO)_3C_3H_5$, when brought in contact with iodine takes up six atoms and forms the *addition product*, di-iodo stearin, $(C_{17}H_{33}I_2COO)_3C_3H_5$. Palmitin, $(C_{15}H_{31}COO)_3C_3H_5$, when similarly treated, forms no addition product, but a small quantity of the *substitution product*, iodo palmitin, $(C_{15}H_{30}ICOO)_3C_3H_5$, and the hydrogen displaced unites with the iodine to form hydriodic acid. The quantity of hydriodic acid thus formed is a measure of the amount of substitution.[1]

1. HANUS'S METHOD.[2]—*Apparatus required.*—Two hundred cubic centimeter white glass bottles with well-ground stoppers; burettes; ten cubic centimeter and fifty cubic centimeter graduates; Erlenmeyer flasks; pipettes; flat watch-glass; short rod; No. 1 beaker.

Reagents.—A solution of iodine (13.2 grams per liter) in glacial acetic acid, to which three cubic centimeters of bromine has been added,[3] $\frac{N}{10}$ sodium thiosulphate, potassium iodide (1 to 10), potassium bichromate (3.8747 grams per liter), starch paste (1 to 200), hydrochloric acid 1.2 specific gravity, chloroform.

[1] McIlhiney, Jour. Am. Chem. Soc., xvi. 275 (1894).

[2] Zeit. Unters. Nahr u. Genussm, iv. 913 (1901); abstr. Chem. Centralb., ii. 1217.

[3] Hunt, Jour. Soc. Chem. Ind., xxi. 454 (1902).

Manipulation.—From 0.12 to 0.15 gram of a drying oil, 0.2 to 0.3 gram of a non-drying oil, or 0.6 to 0.7 gram of a solid fat, are accurately weighed into the two hundred cubic centimeter bottle. This is best effected by pouring out about five grams of the oil into the No. 1 beaker containing the short stirring rod, and setting it into the watch-glass upon the pan of the analytical balance. The whole system is weighed, the beaker removed, and several drops of oil transferred to the bottle by dropping down the rod, being careful that no oil touches the neck. Eight drops are approximately 0.2 gram. The beaker is replaced in the watch-glass and the system again weighed, the difference in weight being the amount of oil in the bottle.

The oil is dissolved in ten cubic centimeters of chloroform, thirty cubic centimeters of the iodine solution added,—best from a burette,—and allowed to stand with occasional shaking for fifteen minutes; with oils of an iodine number of less than 100, ten minutes suffices. Fifteen cubic centimeters of potassium iodide solution are added and the solution titrated, with or without the addition of starch, with sodium thiosulphate until the halogen disappears.

At the same time at which the oil was prepared two "blanks" should be prepared similarly in every

way to the actual tests, except in the addition of the oil, and treated in every respect like them; the strength of the thiosulphate solution should also be determined the same day on which this test is carried out.

Standardization of the Thiosulphate Solution.—Ten cubic centimeters of potassium iodide and one hundred cubic centimeters of water are poured into the Erlenmeyer flask; twenty cubic centimeters of the bichromate solution [1] are now measured in with a pipette, and to this five cubic centimeters of strong hydrochloric acid added and the mixture shaken for three minutes. It is now titrated with the thiosulphate solution until the yellow color of the iodine has almost disappeared; starch paste is now added, and the titration continued until the deep blue color of the solution changes to a sea-green,—due to $CrCl_3$,—which is usually brought about by the addition of a single drop.

The reactions involved are:

$$K_2Cr_2O_7 + 14HCl = 2CrCl_3 + 2KCl + 7H_2O + 3Cl_2;$$
$$3Cl_2 + 6KI = 6KCl + 3I_2;$$
$$6Na_2S_2O_3 + 3I_2 = 3Na_2S_4O_6 + 6NaI.$$

Notes.—Wijs [2] uses iodine chloride instead of bromide; it is more troublesome to prepare and

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[1] Equivalent to 0.2 gram of iodine.

[2] Berichte, xxxi. 752 (1898).

gives results about 1.2 points higher.[1] Either of these methods have the advantage over Hübl's,—first, that the solutions keep better, remaining practically unchanged for several months; secondly, that the action is about sixteen times as rapid, it being completed in fifteen minutes; thirdly, that the solutions are cheaper.

Acetic acid cannot be displaced by carbon tetrachloride as a solvent, as the last traces of iodine are difficult to remove from it. The acetic acid used should be at least 99.5 per cent. and show no reduction with potassium bichromate and sulphuric acid.

2. Hübl's Method.—*Apparatus required.*—As with the preceding, using three hundred cubic centimeter bottles.

Reagents.—A solution of iodine (twenty-five grams per liter) and mercuric chloride (thirty grams per liter), which has been prepared by mixing a solution of each at least twenty-four hours beforehand; other solutions except this the same as with the Hanus method.

Manipulation.—The oil is weighed out as in 1, except that about twenty-five per cent. more may be used.

The oil is now dissolved in ten cubic centimeters of chloroform, thirty cubic centimeters of the iodine

[1] Tolman and Munson, Jour. Am. Chem. Soc., xxv. 244 (1903).

and mercuric chloride solution added, the bottle placed in a dark closet, and allowed to stand, with occasional gentle shaking, for four hours. If the solution becomes nearly decolorized after two hours, an additional quantity should be added. One hundred cubic centimeters of distilled water and twenty cubic centimeters of potassium iodide are added to the contents, and the excess of iodine titrated with sodium thiosulphate. If at this point a red precipitate (HgI_2) is formed, more potassium iodide should be added. As the chloroform dissolves some of the iodine, the titration can proceed until the chloroform layer is nearly colorless, then the starch solution is added, and the operation continued to the disappearance of the blue color.

"Blanks" should be titrated as with the foregoing process, page 53.

Notes.—The method was proposed by Cailletet in 1857, made use of by Mills and Snodgrass[1] in 1883, using, however, bromine and carbon bisulphide, and described in almost its present form by Hübl.[2] The chief factors in its execution are, (1) strength of the iodine solution; (2) the quantity used; and (3) the length of its time of action.

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[1] Jour. Soc. Chem. Ind., ii. 435 (1883).

[2] Dingler Polyt. Jour., ccliii. 281; also Jour. Soc. Chem. Ind., iii. 641 (1884).

1. *The Strength of Iodine Solution.*—According to Hübl's original memoir, the solutions can be kept indefinitely when mixed.

Fahrion[1] states that the solution deteriorated as much as from seventeen to twenty-three per cent. in eight days. Ballantyne[2] confirms the deterioration, but finds it much less, five to eight per cent. in thirty-eight days. This weakening of the solution is probably due to the hydriodic acid formed by the action of the iodine upon the alcohol.[3]

The mercuric chloride acts apparently as a carrier of iodine, as the reaction takes place very slowly without it. (Gantter.[4]) Waller[5] finds that the addition of fifty cubic centimeters HCl, specific gravity 1.19, to the mixed iodine solution preserves it for months. Of the other metallic chlorides, $CoCl_2$ gives the highest true iodine value, $MnCl_2$, $MnBr_2$, and $NiCl_2$ cause practically no addition. (Schweitzer and Lungwitz.[6])

2. *The Quantity of Iodine Solution used.*—The mixed iodine solution as made up should require about fifty-three cubic centimeters of the thiosul-

[1] Jour. Soc. Chem. Ind., xi. 183, abstr. (1892).

[2] Ibid., xiii. 1100, abstr. (1894).

[3] Ibid., xiv. 130 (1895).

[4] Ibid., xii. 717, abstr. (1893).

[5] Chem. Zeitung, xix. 1786, 1831 (1895).

[6] Jour. Soc. Chem. Ind., xiv. 1031 (1895).

phate. Before using, a rough titration should be made, and if it be much weaker than this, a proportionately larger amount added. The action of a large excess of iodine is to increase the substitution rather than addition; increase in temperature or in time produces the same effect.[1]

The excess of iodine recommended is from one hundred and fifty to two hundred and fifty per cent.; some observers recommend from four hundred[2] to six hundred per cent.[3]

3. *Length of Time.*—Two hours is sufficient for olive oil, tallow, and lard, while for linseed oil, balsams, and resins twenty-four hours should be allowed.[4]

Waller[5] thinks that the "iodine number" is really the sum of changes in the fat due to absorption of iodine, oxygen, and chlorine.

The two latter come from the interaction of the iodine and mercuric chloride, setting free chlorine, which sets free some oxygen from the water.

Schweitzer and Lungwitz[6] obtain what they term "the true iodine value" by acting upon the oils for

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[1] Jour. Soc. Chem. Ind., xii. 717, abstr. (1893).

[2] Ibid., xiv. 1031 (1895).

[3] Holde, Mitt. k. t. Versuchs., ix. 81 (1891).

[4] Dieterich, Jour. Soc. Chem. Ind., xii. 381, abstr. (1893).

[5] Analyst, xx. 280, abstr. (1895).

[6] Jour. Soc. Chem. Ind., xiv. 1031 (1895).

twenty-five minutes at 45° C. with iodine dissolved in carbon bisulphide and in the presence of a considerable quantity of mercuric chloride. Practically no hydriodic acid is formed under these conditions, and yet in the case of oleic acid, it absorbs more than the theory requires.

They have studied further the effect of various solvents for iodine instead of ethyl alcohol, as methyl alcohol, ether, carbon tetrachloride and bisulphide.

Ingle[1] has shown that the free acid formed during the process is due to the action of water upon the iodochlorides. Some of these are reduced by potassium iodide with liberation of iodine and consequent reduction in the iodine absorption. Iodine chloride is the active agent, and not hypoiodous acid.

Gill and Adams,[2] using a solution of iodine and mercuric iodide in absolute methyl alcohol, have diminished the amount of substitution that takes place. Oleic acid added the theoretical amount of iodine, and even stearic acid about seven per cent.

For the calculation of the percentage of adulteration of one oil by another Hübl gives the following formula:[3]

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[1] Jour. Soc. Chem. Ind., xxi. 587 (1902).

[2] Jour. Am. Chem. Soc., xxii. 13 (1900).

[3] Dingler Polyt. Jour., ccliii. 281.

"Let x = percentage of one oil and y = percentage of the other oil, further, m = iodine value of pure oil x, n of pure oil y, and I of the sample under examination, then

$$x = \frac{100\,(I-n)}{m-n}."$$

He further states that the age of the oil, provided it be not rancid or thickened, is without influence on the iodine value. Ballantyne[1] finds that light and air diminish the iodine number.

As might be expected, the iodine value is inversely proportional to the cold test.

The method, as will be seen, is a conventional one, and the best results will be obtained by using measured quantities of reagents and carrying through the process in the same manner every time.[2]

The calculation is perhaps most easily made as follows: Subtract the number of cubic centimeters of thiosulphate used for the titration of the oil from that obtained by titrating the blank,—this gives the thiosulphate equivalent to the iodine absorbed by the oil. Multiply this number (of cubic centimeters) by the value of the thiosulphate in terms of iodine, and

[1] Jour. Soc. Chem. Ind., x. 31 (1891).

[2] If, for example, the water be added before the iodide solution, the iodine number is changed by 0.3 per cent.

the result is the number of grams of iodine absorbed by the oil; this divided by the weight of oil used and multiplied by one hundred gives the iodine number.

In case it be desired to recover the iodine used, reference may be had to an article by Dieterich, abstracted in the Jour. Soc. Chem. Ind., xv. 680 (1896).

Bromine Number or Value.—The iodine method just described has, among others, the disadvantage that it fails to distinguish between addition and substitution; this is sometimes of importance, and to accomplish it McIlhiney[1] makes use of the bromine absorption.

Apparatus required.—As in the iodine number.

Reagents.—In addition to some of these used in the iodine number, a solution of $\frac{N}{3}$ bromine in carbon tetrachloride, potassium iodate 2 : 100, carbon tetrachloride.

Manipulation.—From 0.2 to 0.3 gram of a drying oil, 0.4 to 0.5 of a non-drying oil, or 1.0 to 1.2 gram of a solid fat, are accurately weighed into the three hundred cubic centimeter bottle, as in the iodine number (page 53).

The oil is dissolved in ten cubic centimeters of carbon tetrachloride, and twenty cubic centimeters of the bromine solution added, best from a burette.

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[1] Jour. Am. Chem. Soc., xxi. 1084 (1899).

After allowing it to stand two minutes by the watch, twenty or thirty cubic centimeters of potassium iodide are added, in the manner described below, the amount depending upon the excess of bromine. To prevent loss of bromine and hydrobromic acid, a short piece of thin and wide rubber tubing—"bill tie tubing"—is slipped over the lip of the bottle, thus forming a well around the stopper; some of the iodide solution is poured into this and the bottle cooled in cracked ice. Upon removing the stopper the solution is sucked into the bottle, it is shaken to insure the solution of the vapors, and the remainder of the reagent added. The iodine liberated is titrated by sodium thiosulphate in the usual way.

When this titration is finished, five cubic centimeters of the potassium iodate solution are added and the titration repeated. The iodine liberated in this reaction is equivalent to the hydrobromic acid present. Blank determinations should be made with the reagents used, as with the iodine number.

Notes.—Oftentimes, particularly with resins, emulsification of the solution takes place, masking the end point. This can be prevented by the addition of fifty or a hundred cubic centimeters of a ten per cent. solution of salt.

In case ice be not at hand, the vapors will prob-

ably be completely absorbed by passing through the iodide solution in the rubber well.

The reactions involved, in addition to those on pages 52 and 54, are:

Palmitin.

$$(C_{15}H_{31}COO)_3C_3H_5 + 3Br_2 = (C_{15}H_{30}BrCOO)_3C_3H_5 + 3HBr.$$

$$3HBr + 3KI = 3KBr + 3HI.$$

$$6HI + KIO_3 = 3I_2 + 3H_2O + KI.$$

The calculation is similar to that followed in the iodine number (page 60).

The percentage of bromine found as hydrobromic acid is called the bromine substitution figure, and the total percentage absorbed, less twice the bromine substitution figure, gives the bromine addition figure.

The method has the further advantages that it is rapid, the bromine solution is permanent and inexpensive. For data upon various oils, see Table XII.

Saponification Value.—This is expressed by the number of milligrams of potassium hydrate necessary to saponify one gram of the oil. It is called from the originator "Koettstorfer[1] number or value," also "Saponification number," and must not be confounded with "Saponification *equivalent*" as proposed by Allen,[2] which is the number of

[1] Koettstorfer, Fres. Zeit., xviii. 199 (1879).

[2] Commercial Organic Analysis, ii. 40.

grams of oil saponified by 56.1 grams of potassium hydrate.

Apparatus required.—Two hundred cubic centimeter Erlenmeyer flasks; burettes; one inch funnels.

Chemicals required.—Approximately $\frac{N}{2}$ alcoholic potash, $\frac{N}{2}$ hydrochloric acid, phenolphthalein 1 to 500.

Manipulation.—One to two grams of the oil are weighed out into the flask (as in the Iodine value, *q. v.*, page 53) and saponified by twenty-five cubic centimeters $\frac{N}{2}$ alcoholic potash accurately measured from a burette, by heating upon a water-bath, a funnel being inserted in the flask.

When the saponification is complete, shown by the homogeneity of the solution, a few drops of phenolphthalein are added and the excess of alkali titrated with $\frac{N}{2}$ hydrochloric acid. At the same time two blank determinations of the strength of the $\frac{N}{2}$ potassium hydrate must be made.

Notes.—Many prefer to cork the flasks tightly and tie down the stoppers, thus saponifying under pressure; others make use of a return flow condenser, oftentimes merely a long glass tube.

Smetham[1] adds twenty cubic centimeters of ether and finds that it aids saponification. Henriques[2]

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[1] Analyst, xviii. 193 (1893).

[2] Zeit. angew. Chemie, 721 (1895).

uses three to four grams of oil, twenty-five cubic centimeters of petroleum ether, and twenty-five cubic centimeters of *normal* alcoholic potash, saponifying in the cold by allowing to stand over night; the advantage consists in preventing the change in the solution by boiling.

McIlhiney[1] has applied the process to dark-colored substances by making use of the principle that when ammonium chloride is added to a neutral soap solution and the mixture distilled, the amount of ammonia freed is equivalent to the quantity of alkali combined with the fatty acids. As a description of the process is beyond the scope of the present volume, reference must be had to the original article.

As ordinarily prepared, the alcoholic potash solution turns rapidly reddish brown, so that it is very difficult to note the end point. This trouble can be partially avoided by adding a drop or two of the solution to the diluted indicator contained upon a tile after the manner of the titration of iron by bichromate. As the color is probably due to the polymerization of the aldehyde formed by the oxidation of the alcohol, a more satisfactory procedure is to treat several liters of the alcohol with ordinary

[1] Jour. Am. Chem. Soc., xvi. 409 (1894). For a discussion of the theory of the process, see Lewkowitsch, Jour. Soc. Chem. Ind., xvii. 1107 (1898).

potassium hydrate and allow it to stand for a week or ten days. The solution is then distilled—using bits of ignited and granulated pumice to prevent bumping—and the standing solution made from this, using the so-called "potash by alcohol."

The writer has found, if the stock solution be kept under an atmosphere of hydrogen, that the coloration by standing is almost entirely prevented.

Detection of Unsaponifiable Oils.—The qualitative detection takes place by observing the behavior of the solution obtained by boiling the oil with alcoholic potash when diluted with warm water. Any unsaponifiable material will manifest itself as oily drops in the clear alcoholic solution, or as a whitish cloud on the addition of water.

The quantitative determination may take place in two ways: 1. From the saponification number. 2. By gravimetric methods.

1. *From the Saponification Number.*—By Table VIII. it will be noticed that, except for Castor, Rape, and Sperm oils, the saponification number averages 193. If the number found be divided by this figure, the percentage of saponifiable matter will be obtained; this subtracted from 100 will give the unsaponifiable matter. This method gives no idea of the kind of saponifiable matter.

2. *By Gravimetric Methods.*—The procedure is

essentially that of Spitz and Hönig:[1] Ten grams of the oil are boiled fifteen minutes at a return-flow condenser with fifty cubic centimeters of five per cent. alcoholic potash;[2] forty cubic centimeters of water are added and the boiling repeated. The liquid is allowed to cool, washed into a separatory funnel with fifty per cent. alcohol and fifty cubic centimeters of 86° gasolene, thoroughly shaken and allowed to stand. The gasolene layer should separate clearly and quickly from the soap solution and the latter is drawn off; the gasolene is washed two or three times with fifty per cent. alcohol to extract any soap, and these washings added to the soap solution. This latter is extracted until upon evaporation the gasolene leaves no stain upon paper, care being taken to wash the gasolene extracts each time with fifty per cent. alcohol; three extractions with gasolene are usually sufficient.

The gasolene is distilled from these extracts, the residue heated until the gasolene odor disappears, and weighed. From the appearance of the residue some idea of the kind of unsaponifiable matter can be obtained. This in the case of sperm oil will be mainly solid alcohols, probably of the ethylene series.

[1] Zeit. f. Ang. Chem., xix. 565 (1891).

[2] The potash is made by dissolving purified potash in the smallest possible quantity of water and adding absolute alcohol.

Notes.—Care should be taken to use gasolene which leaves no residue on evaporation at 100° C. In case difficulty is experienced in separating the soap and gasolene layers, due to emulsification, especially in the case of wool greases, it can be considerably remedied by adding seven per cent. of common salt to the solution.

Special Tests for Certain Oils.—Lewkowitsch[1] states that little reliance can be placed upon the color reactions of the various oils, an opinion in which the writer can cordially concur; with the exception of the Bechi, Baudouin, and Halphen tests, in the majority of cases with a doubtful sample the doubt will still exist after the color test has been performed.

Bechi's Test for Cotton-seed Oil.—This depends upon the supposition that a substance of an aldehydic nature which reduces silver nitrate is contained in the oil. The method is essentially that of Milliau.[2]

Apparatus.—No. 6 dish; large test-tube; long stirring rod; graduate.

Reagents.—NaOH, 36° Bé., 1.3 specific gravity, thirty per cent. solution; ninety-two per cent. alcohol free from aldehyde; H_2SO_4 1 to 10; litmus paper; three per cent. $AgNO_3$ solution.

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[1] Jour. Soc. Chem. Ind., xiii. 617 (1894).

[2] Jour. Am. Chem. Soc., xv. 164 (1893).

Fifteen grams of oil are weighed into the porcelain dish, using the coarse scales, and heated for about ten minutes upon the water-bath; a mixture of ten cubic centimeters of the caustic soda and ten cubic centimeters of the alcohol is slowly poured upon the oil. The whole is occasionally stirred until the mass becomes clear and homogeneous, and one hundred and fifty cubic centimeters of hot distilled water slowly added so as not to decompose the soap, and the boiling continued until the alcohol is expelled. The dilute sulphuric acid is added to acid reaction, and the separated fatty acids washed three times by decantation with cold water. A portion of these are brought into the large test-tube, fifteen cubic centimeters of alcohol and two cubic centimeters of the silver nitrate solution added, the tube wrapped with brown paper, held in place by an elastic band, and heated, with constant stirring, in the water-bath until one-third of the alcohol is expelled, which is replaced by ten cubic centimeters of water. This heating is continued for a few minutes longer and the coloration of the insoluble fatty acids observed. The presence of cotton-seed in any appreciable proportion causes a mirror-like precipitate of metallic silver, which blackens the fatty acids of the mixture.

Notes.—The alcohol should be proved free from

aldehyde by a blank test. Unless the mixture in the test-tube be thoroughly stirred while heating, it will "bump" and eject the contents. Other methods of procedure consist in applying the test to the oil itself, often after treatment with dilute caustic soda and nitric acid. (Wesson.[1]) The writer had a case in which the *oil* gave the test while the *fatty acids* gave no blackening, showing there was something in the oil itself other than cotton-seed oil which reduced the silver nitrate. The students have no difficulty in detecting a five per cent. adulteration with cotton-seed oil.

Dupont[2] thinks that the reduction of silver nitrate is due rather to sulphur compounds contained in the oil; by passing steam over the oil he obtained a product containing sulphur and the oil still gave the Bechi test. This work has been repeated and confirmed by the author.[3] It is to be noted that while the fatty acids blacken silver nitrate they do not color cadmium, lead or copper salts, but reduce mercury compounds. No indication of an aldehyde was noted by the fuchsine or ammonia tests. The

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[1] Jour. Am. Chem. Soc., xvii. 723 (1895).

[2] Bull. Soc. Chem., (3) xiii. 696; abstr. Jour. Soc. Chem. Ind., xiv. 811 (1895); also Charabot and March, Bull. Soc. Chim., xxi. 252 (1899).

[3] Gill and Dennison, Jour. Am. Chem. Soc., xxiv. 397 (1902).

supposition that the reducing substance is aldehydic in its nature finds support in the fact that if the oil be heated to 240° [1] or be kept for some time [2] it loses this peculiar property.

By purifying the acids by the lead salts Tortelli and Ruggeri [3] are able to detect as little as ten per cent. of heated cotton-seed oil.

Halphen's Test for Cotton-seed Oil.[4]—This depends upon the observation that this oil contains an unsaturated fatty acid which combines with sulphur, giving a colored compound.[5]

Apparatus.—Large test-tube, return flow condenser (long tube), brine bath.

Reagents.—Amyl alcohol and a 1.5 per cent. solution of sulphur in carbon bisulphide.

Procedure.—Ten cubic centimeters of the oil or melted fat are heated with an equal volume of amyl alcohol and of the carbon bisulphide solution of sulphur, at first with frequent agitation, in a steam bath, and then, after the violent boiling has ceased, in the brine bath (105°–110°) for forty-

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[1] Holde, Jour. Soc. Chem. Ind., xi. 687 (1892).

[2] Wilson, Chem. News, lix. 99 (1889).

[3] Jour. Soc. Chem. Ind., xx. 758 (1901).

[4] Halphen, Jour. Pharm. et Chim. (1897), 390.

[5] Raikow, Chem. Zeit., xxiv. 562, 583 (1900).

five minutes to three hours, according to the quantity of adulterant present, the tube being occasionally removed and shaken. As little as one per cent. will give a crimson wine coloration in twenty minutes.[1]

Notes.—If the mixture be heated for too long a time a misleading brownish-red color due to burning is produced. The reaction seems to be peculiar to this oil; it is more sensitive with fresh than old fats, and while, by comparison with a blank, one-sixteenth of one per cent. is noticeable, one-fourth of one per cent. is easily detected. Cotton-seed oil which has been heated to 250° does not give the test; the oil is then not available as food. Heating to 200° does not interfere with the test.[2]

The test is not given by an oil which has been oxidized with sulphuric acid and potassium permanganate, although such an oil gives the Bechi test.[3] This shows that the two tests are not produced by the same substance.

Lard from hogs fed on cotton-seed meal shows this reaction strongly, as if it were twenty-five per

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[1] Oilar, Am. Chem. Jour., xxiv. 355; abstr. Anal., xxvi. 22 (1901).

[2] Soltsien, Zeit. f. off. Chem., v. 135 (1899); Jour. Soc. Chem. Ind., xviii. 865.

[3] Raikow, loc. cit.

cent. oil.[1] The butter from cows similarly fed also yields the reaction.[2]

The test may be applied to the soaps or fatty acids, provided they are not too deeply colored.

The amyl alcohol cannot be omitted nor substituted by ethyl alcohol without impairing the delicacy of the test.[3] The compound in the oil cannot be removed by treatment with animal charcoal.[4]

Baudouin's, or really **Camoin's, test** for Sesamé Oil.

Villavecchia and Fabris[5] apply the test as follows: 0.1 gram sugar is dissolved in ten cubic centimeters of hydrochloric acid of specific gravity 1.18 in a test-tube and twenty grams of the oil to be tested added, the whole thoroughly shaken and allowed to stand. In the presence of one per cent. of sesamé oil the aqueous liquid will be colored red,[6] due to the action of the furfurol formed upon the oil. They state that as olive oils of undoubted purity have shown the reaction in the aqueous layer

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[1] Soltsien, Zeit. f. off. Chem., vii. 140 (1901).

[2] Wauters, Jour. Soc. Chem. Ind., xix. 172 (1900).

[3] Soltsien, loc. cit., 25; Oilar, loc. cit.

[4] Utz, Chem. Rev. Fett. u. Harz.-Ind., ix. 125 (1902).

[5] Zeit. angew. Chemie, 509 (1892); abstr. Jour. Soc. Chem. Ind., xii. 67; also Kerp, Analyst, xxiv. 246 (1899).

[6] Ibid., 505 (1893); abstr. Analyst, xix. 47.

and not in the oily stratum, the color should be looked for in the latter.

The sugar may be replaced by 0.1 cubic centimeter of a two per cent. solution of furfurol and half the quantity of oil used.

Milliau[1] saponifies as in the Bechi test and dries the acids at 105°. Lewkowitsch[2] states that this is a needless complication. Da Silva[3] states that this test has given colors with certain Portuguese olive oils. Kreis[4] states that the active or color-giving constituent is probably phenolic in its nature.

Bach's Test.—According to O. Bach,[5] the acids obtained from rape-seed oil are completely insoluble in David's alcoholic acetic acid, in the proportion of one to fifteen, by volume; those from cottonseed, peanut, sesamé, and sunflower oil dissolve on heating. Those from the last oil separate as a granular precipitate at 15°, while from the other three they gelatinize. The acids from olive oil are completely soluble at the ordinary temperature. David's acid is made by mixing twenty-two cubic

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1 Jour. Am. Chem. Soc., xv. 162 (1893).

2 "Oils, Fats, and Waxes," p. 319.

3 Jour. Soc. Chem. Ind., xvii. 275 (1898).

4 Chem. Zeit., xxvii. 316 (1903).

5 Allen, "Commercial Organic Analysis," vol. ii., pt. 1, p. 128 (1899).

centimeters of fifty per cent. acetic acid (by volume) with thirty cubic centimeters of alcohol, sp. gr. 0.817, 92.07 per cent. (by weight).

Free Acid Test.—*Apparatus required.*—Burettes; one hundred and fifty cubic centimeter Erlenmeyer flasks; thermometer.

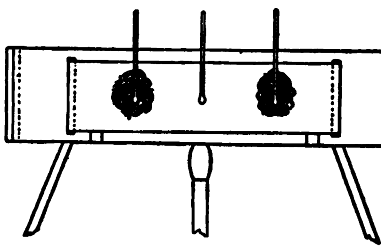
Chemicals required.—$\frac{N}{8}$ Potassium hydrate; ninety-two per cent. alcohol neutralized with sodium carbonate.

About ten grams of the oil are weighed to centigrams into the flask, sixty cubic centimeters of the alcohol added, the mixture warmed to about 60° C., and titrated with the potassium hydrate, using phenolphthalein, the flask being frequently and thoroughly shaken. The result is conventionally reported in per cent. of oleic acid, 1.0 cubic centimeter, $\frac{N}{8}$ KOH is equivalent to 0.047 gram oleic acid.

Spontaneous Combustion Test.—"The apparatus (Fig. 6) consists of a piece of six-inch steam-pipe two feet long, closed at each end by disks of wood; a four-inch tube of thin sheet iron open at each end is contained in this, leaving an inch air-space around it and three inches at the ends. Both tubes are perforated for the insertion of three thermometers. The apparatus is heated by a Bunsen burner placed midway between the ends. The entire apparatus may be enclosed in a shield if the place be drafty. The diameter of the inner tube permits the use of fifty grams of cotton-waste (such as is

used to clean machinery), to which is added an equal weight, fifty grams, of the oil to be tested. The oil is evenly distributed by careful manipulation, the waste rolled compactly but not too tightly, and pushed into the end of the tube, which it should fill so that gentle pressure is required to move it. The bulb of the thermometer is now carefully in-

FIG. 6.



Spontaneous combustion apparatus.

serted into the middle of this ball and the disks inserted. A blank of unoiled waste is placed under the same conditions in the other end. The thermometer in this blank should not be permitted to rise above 100° or 101° C. at the most. To have it reach this temperature the middle thermometer must be kept at about 125°. Since the balls of waste are equidistant from the source of heat, the necessary conditions are fulfilled if there are no drafts to blow the flame or cool one end of the cylinder.

"The thermometers should be read every twenty

minutes, noting each reading down in the proper column, together with the times; a rise in the sample is usually visible in two hours, reaching a maximum in four. An oil which in this time shows a rise to 185° C. is considered as likely to produce spontaneous combustion under very favorable circumstances, while an oil giving a rise to 200° C. is considered to be dangerous.

"The results of the greatest practical value obtained in the use of this apparatus have been, first, determining the cause of fires; and, second, determining the degree of safety of the various oils used in manufacturing. Mineral oil, as is well known, is not liable to spontaneous combustion; and a certain percentage of animal or vegetable oil may be added to mineral oil without materially increasing the danger under ordinary circumstances. This percentage varies according to the oil; with neat's-foot and first quality lard oil some fifty to sixty per cent. may be used, with cotton-seed not over twenty-five per cent. is allowable. The claim so often made for so-called 'safe' oils, said to have been changed by special and secret processes of refining so as to be no longer dangerous, is easily exposed by this test." (Ellen H. Richards, Tech. Quarterly, iv. 346, 1891.)

Notes.—The inner tube is shown in the figure as

being closed by caps; if these fit too closely no opportunity is offered for the oil to absorb oxygen, and hence no correct idea is obtained of the heating effect produced by the oil.

Trouble is often experienced by beginners in not packing the oiled waste sufficiently tight around the thermometer, thus giving low figures. The oils that are liable to cause spontaneous combustion are, in their order of danger, the drying oils, linseed, the semi-drying oils, as cotton-seed and corn, and the animal oils, as lard, neat's-foot, and certain elain oils.

Under these conditions linseed oil showed a temperature of 225° C. in two hours, bursting into flame when exposed to a draft; cotton-seed and corn gave a rise to 205° C. in two and a half hours, lard 220° C., and neat's-foot 230° C. in four hours.

Experiments seem to indicate that better results can be obtained by packing the cotton in wire-gauze cages, heating these in a tube jacketed with water under about fifteen pounds pressure, and, after the thermometer in the sample had reached 125° C., blowing a stream of air through the apparatus at the rate of about 0.4 cubic foot per minute at a pressure of one hundred and sixty-three inches of water.

Mackey [1] uses a smaller apparatus and a less quantity of oil, only fourteen grams, on seven grams of cotton and heats in a water-bath. From his experiments it would seem that even olive oil is almost a dangerous oil, giving 97° and 98° C. rise in one hour, the danger-line being drawn at 100° C. The method should certainly be submitted to the test of time before its adoption. It would seem that the test is an unusually severe one, the cotton being soaked with the oil, which rarely occurs in practice.

Drying Test.—This is, as would be implied, more especially applicable to the drying oils; there are two ways of applying it, exposure of the oil upon finely divided lead (Livache test) and upon a plate of glass.

Livache Test.[2]—One gram of precipitated lead is spread out in a thin layer on a three-inch flat watch-glass and accurately weighed; 0.5 to 0.6 gram of the oil (twenty to twenty-four drops) are brought upon the lead from a pipette, taking care that the drops do not touch each other, the watch-glass and contents again accurately weighed, and then exposed to light and air at ordinary temperature. It is then weighed from time to time, the maximum

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[1] Jour. Soc. Chem. Ind., xv. 90 (1896).

[2] Compt.-Rend., cii. 1167 (1886).

weight being reached in from eighteen to seventy-two hours. The oil that increases most in a given time is considered to be the best drying oil.

Test upon Glass.[1]—A few drops of the oil are brought upon a glass plate inclined at about thirty degrees from the horizontal. A test of the oil is made from time to time by touching it with the fingers, the time at which it does not soil them being noted as the point when it is dry. Good oil should dry in three days.

Archbutt[2] makes this test as follows: A piece of polished plate-glass seven centimeters square by four millimeters thick is cleaned and counterpoised on the balance; it is then heated for an hour at 200° C. in an air-bath to thoroughly dry it. It is taken out, laid on a non-conductor, allowed to cool for three or four minutes, and the hot glass thinly painted with the oil to be tested by means of a camel's-hair brush. When the glass is cold it is weighed and sufficient oil added to make it up to 0.1 gram. Two glasses are coated with the sample and two with a standard oil, all placed on a level surface in a large air-bath at 50° C. and heated for nine hours; one set of plates is withdrawn, cooled,

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[1] Amsel, Jour. Soc. Chem. Ind., xv. 222 (1896).

[2] Jour. Soc. Chem. Ind., xviii. 347 (1899).

and tested by the finger. Good raw linseed is tacky, when tested by the finger when cold, in nine hours and dry in twelve; corn oil is practically dry in fifteen hours, though slightly tacky; cotton-seed, partially dry in eighteen hours and fully dry in twenty-one. Refined rape oil dried in forty-eight hours, and olive oil was sticky after thirteen days.

Lippert[1] confirms Weger's opinion that the Livache test as here carried out is unreliable and advises the use of copper powder instead of lead. This is known as "cement copper," and is prepared similarly to the precipitated lead.

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[1] Chem. Rev. Fett. u. Harz.-Ind., vi. 65; abstr. Jour. Soc. Chem. Ind., xviii. 693 (1899).

CHAPTER IV.

GENERAL CONSIDERATIONS REGARDING LUBRICANTS.

Method of Examination of an Unknown Oil.

According to the results of the viscosity and friction tests, the least viscous oil is to be given the preference. It should be borne in mind, however, that the heat of the journal diminishes the viscosity: for example, at 60° F., if the viscosity of sperm oil be taken as 100, that of 25° paraffine oil is 123; at 100° F. the latter has diminished to 110, and at 250° F. they are practically equal. On account of this change in temperature, as well as the irregularities of the journals, of the feed, and of pressure, a too thinly fluid oil must not be chosen.

The following considerations will aid in the selection of a suitable oil:

1. The flashing point of the oil should be above 300° F.

2. The oil should have an evaporation test of less than five per cent.

3. On general principles the most fluid oil that will stay in place should be used.

4. The best oil is that which possesses the greatest adhesion and least cohesion. This condition is fulfilled, first, by fine Mineral Oils;[1] second, Sperm; third, Neat's-foot; fourth, Lard.

5. For light pressures and high speeds, Mineral Oils of specific gravity 30.5° Bé., flash point 360° F., Sperm, Olive, and Rape (Thurston adds also Cotton-seed), should be employed.

6. For ordinary machinery, Mineral Oils of specific gravity 25° to 29° Bé., flash point 400° to 450° F., Lard, Whale, Neat's-foot, and Tallow, also heavy Vegetable Oils, should be used.

7. For cylinder oils, Mineral Oils of specific gravity 27° Bé., flash point 550° F., alone and with small percentages (1 to 7) of Animal or Vegetable Oils, are employed; the latter are Degras, Tallow, Linseed, Cotton-seed, and blown Rape.

8. For watches and clocks, clarified Sperm, Jaw, and "Melon" oils should be employed.

9. For heavy pressure and slow speed, Lard, Tallow, and other greases, either by themselves or mixed with Graphite and Soapstone, should be used.

10. For very heavy pressure, solid lubricants, as Graphite and Soapstone, are employed.

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[1] Except at high temperatures. Doolittle, Jour. Am. Chem. Soc., xx. 238 (1898).

11. To resist cold, as, for example, for lubricating air-driven rock-drills, Kerosene has been used.

12. The oil should contain no acid to corrode the shaft or journal; the German railroads[1] permit no more than 0.1 to 0.3 per cent. of acid, calculated as sulphuric anhydride, in their oils. For the action of oils upon metals reference may be had to Table X.

REFERENCES.

MILLS, Jour. Soc. Chem. Ind., v. 148, 149 (1886).

COLEMAN, ibid., 359.

REDWOOD, idem, 121–132.

DENTON, Trans. Am. Soc. Mech. Engrs., ix. 369 (1888); xi. 1013 (1890).

ARCHBUTT and DEELEY, "Lubrication and Lubricants," 50–125.

Method of Examination of an Unknown Oil.

There being no specific tests for the different oils, as in the case of the various elementary substances, the analyst should, in attacking an unknown oil, ascertain all possible facts about it, as the source, the use to which it is put, and the cost. A low-priced oil is not likely to be adulterated with one of higher cost. While the prices fluctuate to a considerable extent, yet the following table, it is believed,

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[1] Aisinmann, Zeit. angew. Chemie, xi. 213; abstr. Jour. Soc. Chem. Ind., xiv. 811 (1895).

represents the average price of the various oils, the highest priced being given first:

1. Almond.
2. Castor.
3. Sesamé.
4. Neat's-foot.
5. Rape.
6. Olive.
7. Sperm.
8. Whale.
9. Peanut.
10. Linseed.
11. Tallow.
12. Lard.
13. Cod.
14. Cotton-seed.
15. Mineral.
16. Rosin.

Certain physical properties may aid in the examination. The *color* is of little assistance, as oils may be colored by the use of the oleates or butyrates of iron or copper. *Fluorescence* is valuable as indicating the presence of mineral oil; this can be shown by placing a few drops of the oil on a sheet of ebonite and observing the bluish color.

The *odor* and *taste*, as has already been stated, may to experts reveal much about the nature of the oil under examination. Marine animal oils are detected, especially when warm, by their strong "fishy" odor, while neat's-foot, tallow, lard, olive, resin, and linseed oils each have a well-marked and easily distinguishable odor. Whale oil has a nutty, and rape oil a harsh, unpleasant taste.

The *specific gravity* should next be noted, the oil being exactly at 15° C. The accompanying table shows the groups into which the oils are divided by this criterion:

.875–.884.	.884–.912.	.912–.920.	.920–.937.	.937–.970.
Sperm.	Oleic Acid.	Almond. Lard. Neat's-foot. Olive. Peanut. Rape. Tallow.	Corn. Cotton-seed. Fish. Linseed. Poppy-seed. Sesamé.	Castor. Blown Oils.

The *elaidin* test (page 46) may be applied next, to allow time for the cake to form; it will be followed by the Valenta (page 45) and Maumené (page 48) tests, all of these being done in duplicate. In making the elaidin test, it is advisable to carry on an experiment under the same conditions with a known sample of lard oil.

The presence of vegetable or animal oils can be shown by the separation of compounds of phytosterol or cholesterol according to Bömer.[1]

One hundred grams of the oil are saponified with two hundred cubic centimeters of four per cent. alcoholic potash, the solution evaporated to dryness, dissolved in forty cubic centimeters of water, and extracted three times with an equal quantity of ether. On evaporation of the ether a crystalline product results, which may be recrystallized from seventy per cent. alcohol. Bömer states that the

[1] Jour. Soc. Chem. Ind., xvii. 954 (1898).

form of the crystals is more to be relied upon than a determination of their melting point. Cholesterol, characteristic of animal fats, crystallizes from alcohol or ether in leaflets or rhomboid tables containing one molecule of water of crystallization. Phytosterol, an isomer of cholesterol, crystallizes also from alcohol with one molecule of water in needles forming stars or bundles. As a further means of identification, some of the esters may be made by boiling for an hour with an excess of acetic or other acid anhydride and their melting points determined.

The following table shows the melting points of these alcohols and their esters:

	Cholesterol.	Phytosterol.
Alcohol	146–147°	136–137°
Acetate	113–114°	120–124°
Benzoate	145°	142–143°
Propionate	97–98°	104–105°

As little as one per cent. of cotton-seed has been found in lard, and four per cent. in any oil have been detected.

For the means of distinguishing between drying and marine animal oils, see Halphen, Jour. Pharm. Chim., xiv. 391 (1901), abstracted Jour. Soc. Chem. Ind., xxi. 74, or Chem. Centralb., lxxii. II. 1097 and 1323.

From the result of the examination up to this time a reasonably good idea of the nature of the oil will have been obtained; the iodine test can now be applied and the percentage of adulteration approximated. The data obtained by the Maumené test and specific gravity determination will serve as checks upon this. In case Sesamé, Cotton-seed, Peanut, or Rosin oils be suspected, the specific tests for them can be made.

The saponification test, unless mineral oil be suspected, need rarely be resorted to; the reason being that it would show practically nothing regarding the nature of the oil. This is evident from Table VIII., of all the oils there given, this constant, excepting Castor (178), Rape (174), and Sperm (135), being about 193. Finally, where the importance of the case will warrant, the analyst is advised to prepare a mixture of the oils, using the proportions indicated by the various tests, and subject it to the more rapid tests, as the Specific Gravity, Viscosity, Maumené, and Bromine Number. It should be borne in mind in making out the report that, excepting in the case of the special tests, the results of *one* test cannot be relied upon to determine the nature of an oil, but the evidence of all the tests here given should be carefully weighed and compared before rendering a final verdict.

PART II.

DERIVATION, DESCRIPTION, AND EXAMINATION OF CERTAIN OILS.

In this part the technology and properties of the more commonly occurring oils will be considered under the following heads:

1. Source and Preparation.
2. Physical Characteristics.
3. Chemical Composition.
4. Analytical Constants.
5. Adulterations and their Effects.
6. Uses.

The analytical data are the average of the results from many sources; in addition to these the highest and lowest results are often given.

The classification of the animal and vegetable oils is that of Lewkowitsch, following in the various groups of oils the order of the iodine values.

A. Oils and Fats. Glycerides.

1. Vegetable Oils.

(1) Drying Oils.
(2) Semi-drying Oils.
(3) Non-drying Oils.

2. Animal Oils.

(1) *Marine Animal Oils.*

α. Fish Oils.

β. Liver Oils.

γ. Blubber Oils.

(2) *Terrestrial Animal Oils.*

B. Waxes. Non-Glycerides.

1. Liquid Waxes.

CHAPTER V.

PETROLEUM PRODUCTS.

CRUDE petroleum varies so much with the locality that any statement about it is only approximate; according to Peckham[1], the products which may be obtained from Pennsylvania petroleum are about as follows:

First. The NAPHTHA DISTILLATE, all that passing over above 60° Baumé, about sixteen and five-tenths per cent.

Second. The BURNING OIL DISTILLATE, that passing over between 60° and 36° Bé., about fifty-four per cent.

Third. The LUBRICATING OIL DISTILLATE, that passing over from 36° to 23° Bé., about seventeen and five-tenths per cent.

Fourth. PARAFFINE, two per cent.

Fifth. COKE AND LOSSES, ten per cent.

The Naphtha Distillate.—This is fractionated as follows:

I. Crude Gasolene, cut at 80° Bé., one and five-tenths per cent.

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[1] S. F. Peckham, "Report on Petroleum," p. 165.

II. C Naphtha, between 80° and 68° Bé., ten per cent.

III. B Naphtha, between 68° and 64° Bé., two to two and five-tenths per cent.

IV. A Naphtha, between 64° and 60° Bé., two to two and five-tenths per cent.

Fraction I. is redistilled and the products caught in a mixture of ice and salt, giving:[1]

1. *Cymogene*, 110° to 100° Bé., Bpt. 32° F., largely butane, used for ice-machines.

2. *Rhigolene*, 100° to 90° Bé., Bpt. 65° F., largely pentane, used as a local anæsthetic.

3. *Petroleum Ether*, Sherwood Oil, "Gasolene," 90° to 80° Bé., Bpt. 100° to 150° F., largely hexane, used for carburetting air in the various "gas-machines" and in the laboratory for oil and fat extraction.

4. *Gasolene*, Canadol, 80° to 75° Bé., Bpt. 150° to 190° F., used for oil extraction on the large scale.

Fraction II. is treated with four ounces of oil of vitriol to the gallon in an agitator with mechanical paddles, washed with caustic soda solution, and distilled with steam, yielding:

5. *Naphtha*, Danforth's Oil, 76° to 70° Bé., Bpt.

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[1] Sadtler, "Industrial Organic Chemistry" (1895), p. 80.

160° to 210° F., used in street lamps, stoves, and torches.

Fractions III. and IV. are treated similarly to II., giving:

6. *Ligroine*, 67° to 62° Bé., Bpt. 160° to 225° F., used in pharmacy, in the laboratory, and in sponge lamps.

7. *Benzine* (deodorized), 62° to 57° Bé., Bpt. 225° to 300° F., used as a substitute for turpentine, for cleaning type, and by dyers and scourers.

The Burning Oil Distillate.—This is fractionated into:

V. Crude Burning Oil, 58° to 40° Bé., forty-two per cent.

VI. "B" Oil, 40° to 36° Bé., seventeen per cent.

Fraction V. is treated with acid similarly to II., washed, and distilled as long as the color is good. Three fractions may be obtained:

8. "Export Oil," 110° F., Fire Test (F. T.), shipped to China and Japan.

9. *Export Oil*, 120° F., F. T., shipped to England.

10. *Headlight Oil*, 150° F., F. T., 50° to 47° Bé., ordinary kerosene.

Fraction VI. is treated similarly to V., and on distillation yields:

11. *Mineral Sperm*, or Lantern Oil, 36° Bé., 250° F., Flash point, and 300° F., F. T., used

for passenger traffic illumination and in lighthouses.

The Lubricating Oil Distillate.—The residues from the burning oil distillate are distilled with superheated steam, various fractions being obtained; these are treated with acid, washed, and redistilled, yielding:

"Neutral" Oils, 38° to 32° Bé., used as "wool oils."

Spindle Oils, 32° to 28° Bé.

Loom Oils, 29° to 27° Bé.

Engine Oils, 27° to 23° Bé.

Cylinder Oils, 28° to 25° Bé.

These fractions are filtered through sawdust and salt to remove water, and when too deeply colored through bone charcoal, after the manner of sugar syrups.

In addition to these distilled oils there is another class, the paraffine oils, which are obtained by chilling and pressing certain distillates, yielding solid paraffine and paraffine oil.

A. OILS AND FATS. GLYCERIDES.

CHAPTER VI.

VEGETABLE OILS.

(1) Drying Oils.

Linseed Oil.—Percentage of oil in seeds 38 to 40.

Preparation.—Linseed oil is prepared from the seeds of the flax-plant by expression or extraction. The oil receives its name according to the locality where the seed is grown. Calcutta, La Plata, and Western are some of the brands in this market; the first being considered to be the best, although sometimes equalled by the last.

Properties.—It is of a golden-yellow color and high specific gravity, the highest of any fatty oil likely to be used as an adulterant. On exposure to the air it absorbs oxygen—often causing spontaneous combustion—and dries to a gummy, insoluble substance, linoxyn.

Composition.—It contains the glycerides of isolinolenic, linolenic, linolic,[1] oleic, stearic, palmitic, and myristic acids.

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[1] Hazura and Grüssner, Monatsheft Chem., ix. 180 (1888).

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.9815–.9371	108°–126° C.	Liquid	170–187.7	187.6–195.2
	90	with solid portion.	167.6	
.934	111°		176	191

Adulterants.—Corn, Cotton-seed, Fish, and Rosin oils.

All these adulterants lower the constants given and diminish its drying power; *Cotton-seed* oil would be shown by the Bechi test (page 68); *Rosin* oil by the low Saponification Value, it being unsaponifiable, by the Liebermann-Storch reaction (page 112), and by the rotary power; *Fish* oil[1] may be detected by its odor when warmed.

Uses.—For preparation of paints and as "boiled" and "bleached oil" for preparation of varnishes; by treatment with sulphur chloride for manufacture of rubber substitutes and for soft soaps.

"*Boiled Oil.*"—By heating the oil—preferably in a steam jacketed kettle—from 130° C. upward, with or without the addition of litharge, red lead, lead acetate, manganese dioxide or borate, the oil becomes oxidized, changes color, and dries much more rapidly.

The constants of boiled oil are as follows:

[1] Often menhaden oil, p. 120.

CONSTANTS.[1]

Sp. Gr. 15° C.	Maumené.	Iodine.	Valenta.
.936–.938	100° C.	164–178	60°–74° C.

Bleached Oil.—This is an oil prepared, by special processes kept jealously guarded, for the use of varnish-makers. It may be prepared by heating linseed oil hotter than in the preparation of "boiled oil," to 260° to 300° C., or by forcing oxygen through the oil.

CONSTANTS.[1]

Sp. Gr. 15° C.	Maumené.	Iodine.	Valenta.
.932–.934	104° C.	160	60° C.

Mastbaum[2] states that pressed linseed oil has a higher iodine value than extracted because the more fluid portion is pressed out, and further, that the iodine value changes with the age of the oil.

Linseed oil for varnish-making and other refined purposes should separate but a small quantity of mucilage when heated to 300° C. The oil should be very rapidly heated to this temperature in a metal vessel, poured into a test-tube, and allowed to cool. A suitable oil shows little or no deposit,

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[1] Gill and Lamb, Jour. Am. Chem. Soc., xxi. 29 (1899).

[2] Mastbaum, Zeit. angew. Chem., xxiii. 719; abstr. Jour. Soc. Chem. Ind., xvi. 150 (1897).

whereas a bad one may separate an amount equal in volume to the oil used.

Chinese Wood Oil.—Percentage of oil in seeds 35 to 40.

Preparation.—This oil, called also Japanese Wood oil or Tung oil, is obtained from the seeds of the Elæococca vernicia, and must not be mistaken for gerjun balsam, also known as wood oil.

Properties.—It is pale yellow to dark brown, of unpleasant taste and odor. On exposure to light it is slowly changed to a solid fat, owing to the conversion of the elæomargarine into its isomer, elæostearine. It dries more rapidly than linseed oil, forming a hard film of little adhesive power to the surface beneath. It does not dry on the surface or in layers, as does linseed.

Composition.—It consists of the glycerides of oleic and elæomargaric acids.[1]

CONSTANTS.

Sp. Gr. 15° C.	Iodine.	Saponification.
.940	163	190–197

When heated above 200° C. it gelatinizes and then neither melts nor dissolves.

1 Cloez. Bull. Soc. Chim., xxvi. 286 (1876); Compt.-rend., lxxi. 649; lxxii. 501.

Uses.—Wood oil cannot be regarded as a substitute for linseed oil; when mixed with it, it has given excellent results, especially for out-of-door work. When heated with driers at about 160° C. it has been used to good advantage in the manufacture of oil varnishes. It is used as a floor varnish, in the manufacture of water-proof materials, and products resembling oilcloth.

Poppy-seed Oil.—Percentage of oil in seeds 40 to 50.

Preparation.—Poppy-seed oil is prepared from the seeds of the common poppy.

Properties.—The "cold drawn" oil is colorless or pale golden yellow, that of the second pressing of a reddish color; the taste is pleasant, and it is practically odorless. It dissolves in twenty-five volumes of cold or six of boiling alcohol.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Iodine.	Saponification.
.924–.937	87° C.	133–143	190–197
.925		133	193

Adulterants.—The chief adulterant is *Sesamé* oil, detected by the lower Iodine Value and Baudouin test.

Uses.—The oil is used as a salad oil and for mixing and grinding artists' colors.

Sunflower Oil.—Percentage of oil in seeds 30.

Preparation.—Sunflower oil is obtained from the seeds of the common sunflower.

Properties.—It is a pale yellow oil of bland taste and pleasant odor.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Iodine.	Saponification.
.924–.926	67°–75° C.	118–133	190–194

Gives Bechi test, but not Halphen or Baudouin test.

Uses.—For adulterating other oils, as olive, as an edible oil, for burning, soap- and varnish-making.

(2) Semi-Drying Oils.

Corn Oil.—Percentage of oil in seeds 6 to 10.

Preparation.—Corn or Maize oil is prepared by expression from the germ of the corn separated in the manufacture of starch,[1] or from the residues from the fermentation of alcohol.[2]

Properties.—The former oil is pale to golden yellow, the latter reddish brown.

[1] Jour. Soc. Chem. Ind., xi. 286 (1892).

[2] Kriegner, Dingler Polyt. Jour., 1895, 39; abstr. Jour. Soc. Chem. Ind., xiv. 287 (1895).

CONSTANTS.

Sp. Gr. 15° C. (.916)	Maumené.	Elaidin.	Iodine.	Saponification.
.9215–.924	56°–88° C.	Pasty.	111–123	188–193
.922	85°		115	191
922	100°		124	188[1]

Adulterants.—Mineral and Rosin oils. These would be detected by the lowering of the constants (except Specific Gravity), and the latter by the Liebermann-Storch reaction.

Uses.—For adulterating other oils, especially linseed and lard,[2] and for painting, burning, lubricating, and soap-making, especially transparent soaps.

Cotton-seed Oil.—Percentage of oil in seeds 25.

Preparation.—Cotton-seed oil is obtained by pressing the seeds of the cotton-plant; when first pressed it is ruby-red or black, and is purified by treatment with caustic soda, carrying down the gelatinous substances and color as "cotton-seed foots." The grades in the market are Summer White and Summer Yellow, and Winter White and Winter Yellow, according to the temperature or season of pressing.

Properties.—It is pale yellow in color, and absorbs oxygen slowly from the air.

Composition.—It contains the glycerides of stearic,

[1] Boiled corn oil.

[2] Its presence can possibly be told by the presence of sitosterol. See article by Gill and Tufts, Jour. Am. Chem. Soc., xxv. 254 (1903).

palmitic, oleic, linoleic acids, and some hydroxyacids not yet investigated. (Hazura, Fahrion.)

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
(.916)	(50°)			
.9216–.930	70°–90° C.	Pasty.	101–117	191–196
.922	76°		108	

Adulterants.—It is rarely adulterated; sometimes Linseed oil is used for this purpose when the price admits of it.

Uses.—For adulterating other oils, as a cooking oil both by itself and when mixed with suet, as "Cottolene," "Cotosuet," etc., and for soap stock; it, however, occasions a browning of the product.

Sesame Oil.—Percentage of oil in seeds 50 to 57.

Sesamé oil, known also as Gingili or Teel oil, is prepared from the seeds of the sesamé-plant.

Properties.—It is odorless, of a yellow color and pleasant taste.

Composition.—It contains the glycerides of stearic, palmitic, oleic, and linolic acids, also other bodies[1] the composition of which is not exactly known, to which the color reaction (page 73) is probably due.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.922–.924	65° C.	Pasty.	108–111	187–194
			107	190

[1] Bömer, Chem. Centralblatt, 70, ii. 729 (1899).

Adulterants.—Cotton-seed, Peanut, Rape, and Poppy-seed.

Cotton-seed oil would be shown by the Bechi test, *Peanut* oil by the low Specific Gravity and isolation of arachidic acid; *Rape* oil would lower all the constants and *Poppy-seed* oil raise them, especially the Iodine (138) and Maumené (87°) Values.

The Baudouin test (page 73) is the characteristic test for the presence of Sesamé oil.

Uses.—It finds application as an edible and burning oil, also in tanning and soap-making.

Rape-seed Oil.—Percentage of oil in seeds 33 to 43.

Preparation.—This oil, otherwise known as Colza oil, is obtained from the seeds of *Brassica campestris* or its varieties, colza or turnip.

Properties.—It is of pale yellow color, peculiar odor, and harsh taste.

Composition.—The glyceridics of stearic, oleic, erucic, and rapic acids are contained in the oil.[1]

The free fatty acids vary from 0.5 to 6.2 per cent.

CONSTANTS.

Sp. Gr. 15° C.	Maumené. (92° C.)	Elaidin.	Iodine.	Saponification.
.911–.917	49°–64°		97–106	171–178
.916	55°	Pasty.	101	174

[1] Reimer and Will, Bericht. d. chem. Gesellschaft, xx. 2388 (1887); also Arachidic, Jour. Soc. Chem. Ind., xvii. 1009 (1898).

Adulterants. — Cotton-seed, Poppy-seed, Hemp-seed, Linseed, and refined Fish oil.

Cotton-seed oil would be indicated by Maumené figure (76) and Bechi test; *Poppy-seed* oil by Iodine Value (138); *Hemp-seed* and *Linseed* by Specific Gravity (.934) and Iodine Value (176); *Fish* oil by the odor and high Iodine Value.

Rape-seed oil is distinguished by its almost complete insolubility in glacial acetic acid (Valenta test) and by its high viscosity.

According to Palas,[1] if colza oil be agitated with rosaniline bisulphite, a rose-red coloration is obtained. Other oils of this and the preceding group are unchanged, with the exception of linseed, which is changed to golden yellow. The reagent is prepared by mixing together in the cold thirty cubic centimeters of a one per cent. solution of fuchsin, twenty cubic centimeters sodium bisulphite, 1.31 specific gravity, two hundred cubic centimeters water, and five cubic centimeters of sulphuric acid. The test is capable of detecting two per cent. of colza oil.

Uses.—It is used as a lubricant and a burning oil; because of the difficulty with which it is saponified it finds little application in soap-making.

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[1] Analyst, xxii. 45; abstr. from La Nature (1897).

Blown Rape-seed Oil.—See under Blown Oils, page 117.

Castor Oil.—Percentage of oil in seeds 50.

Preparation.—Castor oil is obtained from the seeds of the castor-oil plant.

Properties.—It is colorless or pale greenish, of mild taste changing to harsh, especially with the American oils.

Composition.—It contains the glycerides of stearic and ricinoleic acids and an active principle to which it probably owes its cathartic properties.

The free fatty acids vary from 0.7 to 14 per cent.; average about 1.

CONSTANTS.

Sp. Gr. 15°. C.	Maumené.	Acetyl Value.	Iodine.	Saponification.
.961–.973	47° C.	153.4	84	178
.961				

Adulterants.—Blown oils, either Linseed, Rape, or Cotton-seed, and Rosin oils.

These, though but ten per cent. be present, cause a turbidity with absolute alcohol, with which castor oil is miscible in every proportion, as it is with glacial acetic acid. Rosin oil would be shown by the lowering of the saponification value.

Uses.—Castor oil is employed in the manufacture of Turkey red oil, for soap-making, illumination, and in medicine.

(3) Non-Drying Oils.

Almond Oil.—Percentage of oil in seeds 45 to 55.

Preparation.—Almond oil is obtained from the seeds of two varieties of the almond-tree, the sweet and bitter almond, the latter yielding the more oil.

Properties.—It is a bland thin oil of pale yellow color, mainly pure olein.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.914–.920	53° C.	Solid.	93–102	190
.918			97	

Adulterants.—It is adulterated with Peach and Apricot Kernel oils, Cotton-seed, Peanut, Lard, Olive, Sesamé, and Poppy-seed oils.

The first two are well-nigh impossible of detection. *Cotton-seed* oil would be indicated by the Maumené figure (76) and Bechi test. *Peanut* oil would be shown by the isolation of arachidic acid. *Lard* oil by the odor when heated, and also *Olive* by the deposition of stearin when cooled to —5°. *Sesamé* oil could be detected by the Baudouin test and *Poppy-seed* by the Iodine Value (138).

Uses.—Almond oil is used in medicine or whenever a fairly permanent oil is required.

Peanut Oil.—Percentage of oil in seeds 50.

Preparation.—By the cold pressing of the common

peanut a colorless, pleasant-tasting oil is obtained, which is used as a salad oil; a second pressing yields an oil of inferior quality, used as an edible and burning oil; a third pressing at a higher temperature yields a grade employed in soap-making.

Properties.—It varies in color from white to yellow.

Composition.—It contains the glycerides of palmitic, linolic, oleic, arachidic, and lignoceric acids.[1]

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.916–.922	44°–67° C.	Solid.	85–105	189–197
.917	51°		98	194

Adulterants.—Cotton-seed, Rape, Sesamé, and Poppy-seed are used to adulterate this oil.

Cotton-seed oil would be shown by the rise in the melting point of the fatty acids, those of peanut oil melting at about 28°, while those from cotton-seed melt about ten degrees higher; it would further be shown by the Bechi test. *Rape* oil would be indicated by the low Saponification Value (178), *Sesamé* oil by the Baudouin test, and *Poppy-seed* oil by the Specific Gravity (.924) and high Iodine Value (138).

[1] Gossmann and Scheven, Annalen, xciv. 230 (1885); Kreiling, Berichte, xxi. 880 (1888); Caldwell, Annalen, ci. 97 (1857).

Characteristic Test.—The oil can be detected in other oils by the isolation of its peculiar acid,—arachidic acid. This is effected, according to Renard,[1] as follows: The fatty acids are prepared from ten grams of the oil by the usual process of saponification and acidification with hydrochloric acid, dissolved in ninety per cent. alcohol and precipitated with lead acetate. The lead salts are filtered and extracted with ether, leaving lead palmitate and arachidate undissolved. These two salts are treated with hydrochloric acid, and when the liquid has cooled the fatty acids are separated and dissolved in fifty cubic centimeters of hot ninety per cent. alcohol. If peanut oil be present in the sample, a crop of crystals consisting of arachidic (and lignoceric) acid will be obtained; these are filtered off and washed with a measured quantity of ninety per cent. alcohol, also with seventy per cent., in which latter they are less soluble, and they are finally dissolved in boiling absolute alcohol. This solution is evaporated to dryness in a tared flask and the residue weighed; to this is added the quantity dissolved by the ninety per cent. alcohol, of which one hundred cubic centimeters at 15° C. dissolve 0.022 gram, or at 20°, 0.045 gram. Finally, the melting point of the crude acids

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[1] Renard, Compt.-Rend., lxxiii. 1330 (1871); see also Archbutt, Jour. Soc. Chem. Ind., xvii. 1124.

should be determined, which should be 71° to 72°. Peanut oil contains about five per cent. of arachidic acid, hence by multiplying the weight of the acids thus obtained by twenty a rough idea of the percentage present can be obtained.

Uses.—These have been already given under the preparation.

Olive Oil.—Percentage of oil in the fruit 40 to 60.

Preparation.—Olive oil is prepared by expressing or extracting the fruit of the olive-tree; the oil varies greatly according to the tree, there being no less than three hundred varieties in Italy alone, and also the degree of ripeness and manner of gathering of the fruit itself.

Properties.—It varies in color from almost colorless to golden yellow or green.

Composition.—It contains palmitin, stearin, olein, and linolin,[1] the solid glycerides constituting about twenty-eight per cent. of the oil.

The free fatty acids vary from 1 to 24 per cent.

According to Allen, an oil containing more than five per cent. of free fatty acids is unfit for a lubricant, as it attacks the metals, and also, according to Archbutt, as a burning oil, as it causes charring of the wick.

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[1] Hazura and Grüssner.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.914–.918	41°–45° C.	Very solid.	77–88	185–206
.916	35°		82	194

Adulterants.—Cotton-seed, Peanut, Rape, Sesamé, and Poppy-seed.

Cotton-seed oil would be shown by the Bechi test and the high Maumené figure (76) and Iodine Value (108). *Peanut* oil by isolation of Arachidic Acid and high Iodine Value (98). *Rape* oil would be indicated by the Saponification Value (175) and Iodine Value (101). *Sesamé* oil by the Baudouin test. *Poppy-seed* oil by the Iodine Value (138) and Maumené figure (87).

Olive oil is characterized by the low Maumené and Iodine Values and by the solid elaidin.

Uses.—It is used as an edible oil, for oiling textiles, as a soap stock and as a burning oil.

ROSIN OILS AND TURPENTINE.

Rosin Oil.—Rosin oil is prepared by the distillation of common rosin (colophony) in stills holding about thirty barrels. About eighty-five per cent. of rosin oil and three per cent. of rosin spirits, or pinoline, are obtained. Acid water, gas, coke, and losses

account for the remaining twelve per cent. The product obtained is a thick oil, known as "First Run;" this is redistilled, yielding a darker-colored oil, called "Second Run." This operation is repeated, yielding "Third," "Fourth," and even "Fifth Run."

A comparison of their properties is shown in the table below.

PROPERTIES OF ROSIN SPIRITS AND OILS.

ROSIN SPIRITS.

Sp. Gr. at 15°.	Maumené.	Iodine.	Saponification.
0.903	91	12	. .

Free Acid, 15.0.

ROSIN OIL, SECOND RUN.

Sp. Gr. at 15°.	Maumené.	Iodine.	Saponification.
.987	82	59	34

Free Acid, 12.6.

ROSIN OIL, THIRD RUN.

Sp. Gr. at 15°.	Maumené.	Iodine.	Saponification.
.985	84.0	77	25

Free Acid, 18.7.

ROSIN OIL, FOURTH RUN.

Sp. Gr. at 15°.	Maumené.	Iodine.	Saponification.
.981	82.5	23	20

Free Acid, 9.6.

Deodorized Rosin oil is that portion of the later runs which is freed from the "spirits" by fractional distillation.

Uses.—"First Run" is employed in making axle grease, in oiling leather, and making cements. "Second Run" finds use in printing ink and in the leather industry. "Third" and "Fourth Runs" are used mainly for mixing with other oils.

Qualitative Test.—Rosin oil may be detected by the Liebermann-Storch reaction.[1] One to two cubic centimeters of the oil are shaken with an equal quantity of acetic anhydride and gently warmed. When cool, the acetic anhydride is pipetted off and tested by the addition of one drop of concentrated sulphuric acid. A fine violet color is produced in the presence of rosin oil. Cholesterol which is contained in the animal fats produces a similar coloration; this can be removed by saponifying the oil as completely as possible and shaking out the somewhat dilute soap solution with ether or petroleum ether. The soap solution is then acidified, setting free the fatty acids, and these treated with acetic anhydride as if they were the oil.

Renard's test modified by Allen[2] consists in adding a few drops of stannic bromide, dissolved in carbon bisulphide, to a few drops of the oil, also dissolved in carbon bisulphide. Should rosin oil be

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[1] Storch, Jour. Soc. Chem. Ind., vii. 136 (1888).

[2] Commercial Organic Analysis, ii. 463.

present a violet color will be produced, which on standing forms a deposit at the bottom of the tube. Glacial acetic acid is recommended as a solvent in the case of mineral oils, these not dissolving it to any appreciable extent and not masking the reaction.

Wiederhold[1] states that rosin oils are dissolved at 15° C. by half their volume of anhydrous acetone, while mineral oils, especially American, are almost unacted upon by it.

Turpentine.—*Preparation.*—Turpentine is prepared[2] by distilling pine resin in copper stills of about eight hundred gallons capacity; the process requires some care to prevent overheating and obtain a fine quality of rosin. To aid the process, after the crude resin is melted, a stream of tepid water from the condenser is run into the still, thus making a distillation with steam. The yield and quality vary according to the length of time the trees have been producing resin, both growing inferior with age. The crude resin, or "dippings," of the first season is called "virgin dip," and produces the finest quality of rosin, W. W. (water white) and W. G. (window glass); the better grades are N, M, and K, passing

[1] Fres. Zeit., xxxiii. 111 (1894).

[2] Condensed from a monograph on "The Timber Pines of the Southern United States," by Filibert Roth, U. S. Dept. of Agriculture (1896).

through the poorer grades to the black A. From two hundred and twenty-five barrels of soft turpentine and one hundred and twenty barrels of hard gum, the product of a second season, nineteen hundred gallons of turpentine and two hundred barrels of amber rosin, I, H, or G, were produced.

The resin is chiefly obtained from the Long-leaf Pine, *Pinus palustris* or *australis*, known also as Southern, Yellow, or Hard Pine.

Properties and Composition.—Turpentine is a colorless liquid of peculiar taste and odor. On exposure to the air it absorbs oxygen and gradually becomes resinous. It consists mainly of a hydrocarbon, Pinene, $C_{10}H_{16}$.

CONSTANTS.

Sp. Gr. 15° C.	Iodine.
.862–.87	381[1]

The boiling point is 155° to 156° C., and eighty-five per cent. should pass over between 155° and 163°, the remainder below 183°.[2] The flash point is 92° to 98° F. by the Abel tester (about 119° to 125° F. Mass. tester). American turpentine deflects

[1] Wilson, Chem. Trade Jour., vi. 316; Jour. Soc. Chem. Ind., ix. 657 (1890).

[2] Long, ibid., x. 261; Jour. Soc. Chem. Ind., xi. 549.

polarized light to the right, although a sample obtained from spruce-trees had a specific rotation of —40.79.[1]

Adulterants.—Petroleum and Shale Products, Rosin Spirits, and Russian Turpentine are the chief adulterations.

Petroleum and Shale Products: the lighter ones would be indicated by the lowering of the specific gravity, flash test, and iodine value, they having a value of 30 and 70 respectively, and by distillation. Kerosene might be detected by the "bloom." To determine the quantity of petroleum or heavy oil added, Vulpius[2] floats a gram of the suspected sample and of a pure sample each in a separate watch-glass upon a beaker of water kept at 80°. When the pure sample has evaporated both are weighed, the residue from the pure sample deducted from the other, and this difference represents the heavy oil added. According to Burton,[3] the oxidation with fuming nitric acid gives fairly quantitative results on the percentage of petroleum products present. This is effected by dropping slowly one hundred cubic centimeters of the sample into three

[1] Long, Jour. Am. Chem. Soc., xvi. 844 (1894).

[2] Apoth. Zeit., vi. 289; abstr. Jour. Soc. Chem. Ind., x. 800 (1891).

[3] Am. Chem. Jour., xii. 102.

hundred cubic centimeters of fuming nitric acid in a flask immersed in cold water; the oxidation products are dissolved in hot water and the petroleum remains.

Rosin Spirits could be detected by distillation and treatment of the residue with stannic bromide (Renard's test, page 112) dissolved in carbon bisulphide. The addition of rosin spirits might cause a lowering of the iodine value, that for rosin spirits calculated from the bromine absorption being 292 to 322. (Allen.) *Russian Turpentine* would be shown by the higher temperature of distillation, 170° to 180°. Pure turpentine should leave no residue upon writing-paper after half an hour.

Uses.—Turpentine finds extended use as a solvent for fats, waxes, resins, and rubber, and as a "drier" in paints.

Varieties of Turpentine and Sources.—American turpentine, from *Pinus palustris* or *australis*, the Longleaf Pine, dextrorotary.

English turpentine, from gum collected in America from *P. australis* and *P. tæda*, Loblolly.

French, from *Pinus maritima*, Sea-pine, lævorotary.

German, from *P. sylvestris*, Scotch Pine or Fir, *P. nigra*, Black Pine, and *P. rotundata.*

Venice, from *Larix europæa*, Larch.

Russian, from *P. sylvestris*, and *P. ledebourii*, dextrorotary.

Blown Oils.—*Preparation.*—Blown, Base, Thickened or Oxidized oil is usually prepared by heating the oil to 70° or 110° in a jacketed kettle and forcing a current of air through it; after the action is once started no further heating is usually necessary.

Properties.—The color of the oil darkens slightly and the density and viscosity are much increased. Benedikt and Ulzer think that the fatty acids are oxidized to hydroxyacids. The oils submitted to this process are chiefly Rape and Cotton-seed, although it is often applied to Linseed, Sperm, and Seal oils.

CONSTANTS.[1]

Sp. Gr. 15° C.	Maumené.[2]	Iodine.	Saponification.	
.967	253–(57°)	63.6	197.7	Rape.
.974	227	56.4	218.3	Cotton-seed.

Uses.—On account of their high viscosity, blown oils are used to mix with other oils for lubricating purposes.

[1] Thomson and Ballantyne, Jour. Soc. Chem. Ind., xi. 506 (1892).

[2] Specific temperature reaction.

Palm Oil.—*Preparation.*—Palm oil is obtained from the flesh or pericarp of the palm-nut; this grows in immense quantities on the west coast of Africa. The fruits are fermented, whereby the oil rises to the top, or it is expressed from the fresh fruit. The latter process yields the finer and more fluid product.

Properties.—Owing to its method of preparation its properties are quite varied. It is of a buttery or tallowy consistency, of orange-yellow to dirty red in color, and has an odor in some samples recalling that of violets. By heating to a high temperature or treatment with acids it may be bleached.

Composition.—It is mainly palmitin, with some olein and free palmitic acid.

CONSTANTS.

Sp. Gr. 99° C.	Iodine.	Saponification.
.859	50–53.5	196–202

Adulterants.—Water and dirt, mostly sand.

Uses.—For the manufacture of soap and candles and coloring other oils.

Cocoanut Oil.—*Preparation.*—Cokernut oil is obtained from the fat of the cocoanut, the fruit of a species of palm. The finest quality is that prepared in Cochin (Malabar) from the fresh fruit. Infe-

rior varieties are made from the dried kernels, or "coprah," which contains 60 to 70 per cent. of oil.

Properties.—It is a solid white fat of bland taste and peculiar odor, readily turning rancid. It is soluble in two volumes of absolute alcohol at 31° C.

Composition.—It contains a larger proportion of volatile acids than most oils, the glycerides of caproic, caprylic, capric, oleic, lauric, and myristic acids are among those present.

CONSTANTS.

Sp. Gr. 99° C.	Iodine.	Saponification.
.874	8–9	253–268

Adulterants.—It is rarely adulterated.

Uses.—It is used in soap-making (marine soaps), in candle-making, and as an edible fat.

CHAPTER VII.

ANIMAL OILS.

(1) Marine Animal Oils.

a. Fish Oils.

Menhaden Oil.—This oil is otherwise known as mossbunker, pogy, porgy, or whitefish oil.

Preparation.—It is prepared from the menhaden by steaming and expression. There are several grades in the market, differing in appearance according to the source from which they are derived. They are Select Light Strained, Select Light, Choice Brown, Dark, and Gurry oil. The better varieties are obtained by gentle pressure and subsequent bleaching, and the others by the pressing of the residues.

Properties.—It is yellow to brown in color, and oxidizes readily on exposure to the air.

Composition.—It apparently contains the glycerides of linoleic, myristic, asellic, and acetic acids, and also isocholesterol.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.927–.933	123°–128° C.	Liquid.	147.9–160	189–192
.930	126°		154	190

Adulterants.—The chief adulterant is Mineral oil, which would be shown by a lowering of all these constants.

Uses.—It is used in currying, for adulterating other oils, as linseed, whale, and sardine, as a substitute for linseed oil, and as a burning oil for mines.

REFERENCE.

G. B. GOODE, "The Natural and Economic History of the American Menhaden," U. S. Commission of Fish and Fisheries, vol. v., 1879.

β. Liver Oils.

Cod Oil.—Three varieties of cod or cod-liver oil are obtainable in the market, the *pale yellow*, or "steam rendered," and the *light brown*, both of which are used in pharmacy, and for the examination of which recourse must be had to larger works. The other, the *brown* oil or "cod oil," used in currying, may be derived from the liver of any fish, hence it is impossible to give any data upon which judgment may be formed.

γ. Blubber Oils.

Whale Oil.—*Preparation.*—Whale or Train oil is obtained by rendering the blubber of various species of whales except the sperm and bottlenose.

Properties and Composition.—It has a strong fishy odor, a "nutty" taste, and is of a light-yellow to

yellowish-brown color. Little is known regarding its constitution. As may be expected, its composition varies widely.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Iodine.	Saponification.
.925–.930	85°–91° C.	110–130	188–193
.927	88°	120	190

Adulterant.—It is largely adulterated with Seal oil, which there is little chance of detecting.

Uses.—Whale oil is used as a leather dressing, as a burning oil, and to mix with other oils as a lubricant.

(2) Terrestrial Animal Oils.

Neat's-foot Oil.—*Preparation.*—Neat's-foot oil is obtained from the feet of neat cattle. The hoofs are separated, the bones of the foot disjointed, and the latter boiled with water, the emulsion allowed to settle, and the oil which rises separated. As is the case with all oils, that which is obtained by the least degree of heat or pressure is the best.

Properties.—It is of a light-yellow color, bland taste, possesses a peculiar odor, and little tendency to turn rancid.

Composition.—It is nearly pure olein, containing a small quantity of stearin, which it frequently deposits. The free fatty acids may amount to six per cent.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.914–.916	37°–48.5° C.	Solid at times.	56–72	194
	39		67.5	
	42		64–70	

If the iodine number be less than 63, it probably contains hide oil. The fatty acid should be less than one per cent. Titer test may be from 17° to 26°.

Adulterants.—Fish, Poppy-seed, Rape, Cotton-seed, Mineral oils, and other hoof oils.

Fish oil would be shown by the Iodine Value and Maumené test, also by the odor when heated; *Poppy-seed* oil by the Gravity (.925) and Iodine Value (138); *Rape* oil by Saponification (178) and Iodine Value (101); *Cotton-seed* by the Bechi test and Iodine Value (108); *Mineral* oil by the lowering of all the constants given.

Uses.—Neat's-foot oil finds application as a lubricant, either by itself or mixed with other oils, and for currying purposes.

Horse Oil.—Horse oil is prepared by rendering dead horses.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Iodine.	Saponification.
.916–.922	46°–55° C.	75–86	197.1

It is used for mixing with and adulterating other oils, as, for example, neat's-foot; when refined it has been used to adulterate olive oil.

Lard Oil.—*Preparation.*—Lard oil is obtained by pressing lard; upright screw-presses are used and a pressure of about eight thousand pounds to the square inch employed; from forty to sixty per cent. of the lard is obtained as oil.

Brands.—These vary according to the source whence they are derived; the various lards in the American market are: *Neutral Lard*, obtained from the "leaf" by rendering at a low temperature (105° to 120° F.), used in making butterine. Only a portion of the fat is thus extracted; the operation is then completed, yielding *Leaf Lard*. *Choice Lard* is obtained from some parts of the leaf and fat from the backs. *Prime Steam Lard* is the product obtained from the trimmings, head, heart, and some intestinal fat. *Gut Grease* is obtained by rendering all the other parts of the hog except the heart, liver, and lungs.[1]

Besides these products obtained from the live hog, there are *Butchers' Lard* or *Crackling Grease*, obtained from scraps and trimmings, and *White Grease* and *Brown Grease*, which are obtained from hogs dying in transit, being prepared from the eviscerated animal and its viscera respectively.

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[1] Condensed from "Lard and Lard Adulterations," by H. W. Wiley, U. S. Dep't Agriculture, Bull. 13, 1889, p. 14.

Lastly, there is *Yellow Grease*, a product of the refuse of the packing-houses.

All but the first two lards are pressed, yielding an oil which is classed according to its color as "Prime" (very light straw) to "No. 2" (brown).

The varieties in the market are as follows: "Prime" Lard oil, prepared from Prime Steam Lard; "Pure" Lard oil, from No. 1 Lard and White Grease; "Extra No. 1," from Light Yellow Grease; "No. 1," from Yellow Grease; "No. 2," from Brown and Gut Grease; and "Crackling Oil," from Crackling Grease.

Properties.—The color varies from very light straw to brown, and the odor from almost none to offensive in the No. 2 lards.

Composition.—Its chemical composition is largely olein, with admixture of stearin and palmitin.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.914–.916	39° C.	Solid cake.	60	195–6.[1]
	41°		72.5[2]	
	43°		75[3]	

Various parts of the animal give oils which vary considerably; the iodine values of oils from different sources are as follows:[4]

[1] No. 2 lard. [2] No. 1 lard. [3] Prime lard. [4] Wiley, *loc. cit.*

Leaf.	Intestine.	Back.	Foot.	Head.
52.5–53	57.3	60.6	77.3	85

Adulterants.—These are Cotton-seed, Corn, and Neutral Petroleum oils.

Cotton-seed oil would be shown by the Elaidin, Maumené, and Bechi tests. *Corn* oil would be indicated by the Maumené test (58) and Iodine number (115). *Petroleum* by the flash test and lowering of the constants.

Uses.—Lard oil is used as a burning and lubricating oil, as an edible oil, and for oiling textile material preparatory to spinning.

REFERENCE.

Wesson, Jour. Am. Chem. Soc., xvii. 723–735 (1895).

Tallow Oil.—*Preparation.*—Tallow oil is prepared by pressing tallow after the manner of lard, *q. v.*

Properties.—It is a light-yellow bland oil, and of an odor resembling tallow.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.916	35	Solid cake.	56	197

Uses.—It is used to mix with other oils and as a lubricant.

Elain or Red Oil.—*Preparation.*—Elain oil, or, as it is sometimes called, "Saponified Red oil," is obtained by the saponification of the solid fats by the lime, sulphuric acid, or water methods. The fatty acids thus freed from their combination with glycerin are allowed to solidify and are pressed. According to the temperature, more or less stearic and palmitic acids go into the product; these can be separated by distillation.

It is oftentimes semi-solid, resembling tallow; the distilled varieties are light brown to deep red.

Composition.—Chemically speaking, it is nearly pure oleic acid.

CONSTANTS.[1]

Sp. Gr. 15° C.	Free Fatty Acids.	Iodine.	Saponification
.899–.908	80–97	90[2]	200

It may contain some unsaponifiable matter, consisting of hydrocarbons formed in the process of distillation; these may vary from three to seven per cent.

Uses.—It is used for oiling wool, as it readily saponifies, and in soap-making.

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[1] Allen, Lewkowitsch.

[2] Iodine number of the pure acid.

B. WAXES.

1. LIQUID WAXES.

Sperm Oil.—*Preparation.*—The real sperm oil is obtained from the great cavity in the head of the sperm whale; it is often mixed with the oil obtained from the body, or "blubber oil." The process of manufacture consists in chilling the crude oil, separating the spermaceti by pressure, and bleaching the expressed oil in thin layers by exposure to the sun.

Properties.—It is a limpid, pale-yellow oil of faint odor and taste.

Composition.—It contains no glycerides (Allen, Lewkowitsch), but is a mono-ester, a compound of an alcohol and an organic acid. When saponified these alcohols are freed, and the oil yields forty per cent. of unsaponifiable matter. It may contain three-tenths per cent. of free fatty acids.

CONSTANTS.

Sp. Gr. 15° C.	Maumené.	Elaidin.	Iodine.	Saponification.
.875–.884	45°–47° C.	Solid at times.	81–84	123–147
.880			(70)	

Adulterants.—Owing to its high cost it is often adulterated, Whale, Mineral, Rape, Liver, and Arctic Sperm (bottlenose whale) oil being used for this purpose.

Whale oil would be shown by the strong fishy odor and "nutty" taste, also by the raising of all the constants. *Mineral* oils would be indicated by the low flash point, corresponding to a gravity of 0.880, and by the lowering of the constants. *Rape* oil by the high Saponification Value (178) and the isolation of the glycerin, which when multiplied by ten gives the fatty oils. *Liver* oils would be revealed by the violet coloration with sulphuric acid and rise in the constants. *Arctic Sperm* oil might be shown by the taste.

Uses.—It is employed as a lubricant; the viscosity is less than any other non-drying fatty oil, and also varies less than any other oil with increase of temperature.

REFERENCES.

STARBUCK, "History of American Whale Fishery from Earliest Inception to 1875."

Report of U. S. Commissioner of Fisheries, vol. iv., 1875.

SCAMMON, "Mammalia of North-Western America."

APPENDIX.

TABLES, REAGENTS, AND RAILROAD SPECIFICATIONS.

TABLE I.

Requirement of Various States and Cities regarding Flash and Fire Test of Illuminating Oils.

Name.	Flash, ° F.	Fire, ° F.	Instrument.
Arkansas	. .	130	Tagliabue.
Columbia, District of	120	. .	
Connecticut	. .	110	
Florida	. .	130	Tagliabue.
Georgia	. .	120	
Illinois	. .	150	Tagliabue.
Indiana	120	. .	Indiana.
Iowa	105	. .	Elliott.
Kansas	. .	110	Tagliabue.
Kentucky	. .	130	
Louisiana	125	. .	Tagliabue.
Maine	120	. .	Tagliabue open.
Massachusetts	100	. .	Tagliabue open.
Michigan	120	148	Foster.
Minnesota	110	. .	Minnesota.
Missouri	. .	150	Tagliabue.
Montana	110	. .	
Nebraska	100	. .	Foster.
New Hampshire	100	120	Tagliabue.

Name.	Flash, ° F.	Fire, ° F.	Instrument.
New Jersey	100	115	
New Mexico	. .	150	
New York	. .	110	Tagliabue.
North Carolina	100	. .	Foster.
North Dakota	100	. .	
Ohio	120	. .	Foster.
Pennsylvania	. .	110	Tagliabue.
Rhode Island	. .	110	
South Dakota	110	. .	Foster.
Tennessee.	120	. .	Open cup.
Vermont	. .	110	Tagliabue.
Wisconsin	120	. .	Wisconsin.

Requirements of Cities where different from State Law.

Name.	Flash, ° F.	Fire, ° F.	Instrument.
Baltimore, Md.	120	. .	
Denver, Col.	110	. .	Tagliabue open.
Los Angeles, Cal.	110	. .	Tagliabue open.
Meriden, Conn.	. .	125	
Milwaukee, Wis.	110	. .	
Newark, N. J.	. .	110	
New Haven, Conn.	. .	110	Tagliabue open.
New Orleans, La.	. .	110	Tagliabue open.
New York, N. Y.	100	. .	Elliott.
Richmond, Va.	. .	110	Tagliabue.
Sacramento, Cal.	110	. .	Tagliabue open.
San Francisco, Cal. . . .	100	. .	Tagliabue open.
Wilmington, Del.	. .	110	

TABLE II.

Showing the Flash and Fire Test of Various Oils.[1]

Name.	Flash, ° C.	Flash, ° F.	Fire, ° C.	Fire, ° F.
Corn	249	480	335	635
Cotton-seed	305	582	340	644
Prime Lard	264	530 / 600	340	644
No. 2 Lard	215	419	242	468
Boiled Linseed	192	378	300	572
Raw Linseed	274	525	340	644
Neat's-foot	226	439	273	523
Olive	233	451	283	541
25° Paraffine	210	410	246	475
75% 25° Paraffine, 25% Neat's-foot	210	410	244	471
75% 25° Paraffine, 25% Lard	210	410	254	489
50% 25° Paraffine, 50% Lard	218	423	267	513
25% 25° Paraffine, 75% Lard	227	441	284	543
Distilled Red	184	364	213	415
Sperm No. 1	220	428	270	518
Sperm No. 2	252	486	301	574

Flash Point of Certain Organic Compounds.

Alcohol, absolute	12° C.[3]	81° F.[2]
Alcohol, + 0.5 per cent. ether	9	75
Alcohol, 4 per cent. by volume	6–8	181
Benzene, C_6H_6	—8	45
Turpentine	—	119–125

[1] Done with the apparatus described upon page 36.

[2] Raikow Chem. Zeit., 145 (1899): Holde and Pelgry, Chem. Centralb., ii. 546 (1899). Done with Abel's tester and calculated to Massachusetts tester by adding 27° F.

[3] Abel tester.

TABLE III.

Relation of Baumé Degrees to Specific Gravity and the Weight of One United States Gallon at 60° F.

Baumé.	Specific Gravity.	Pounds in Gallon.	Baumé.	Specific Gravity.	Pounds in Gallon.	Baumé.	Specific Gravity.	Pounds in Gallon.	Baumé.	Specific Gravity.	Pounds in Gallon.
10	1.0000	8.33	31	0.8695	7.24	52	0.7692	6.41	73	0.6896	5.75
11	0.9929	8.27	32	0.8641	7.20	53	0.7650	6.37	74	0.6863	5.72
12	0.9859	8.21	33	0.8588	7.15	54	0.7608	6.34	75	0.6829	5.69
13	0.9790	8.16	34	0.8536	7.11	55	0.7567	6.30	76	0.6796	5.66
14	0.9722	8.10	35	0.8484	7.07	56	0.7526	6.27	77	0.6763	5.63
15	0.9655	8.04	36	0.8433	7.03	57	0.7486	6.24	78	0.6730	5.60
16	0.9589	7.99	37	0.8383	6.98	58	0 7446	6.20	79	0.6698	5.58
17	0.9523	7.93	38	0.8333	6.94	59	0.7407	6.17	80	0.6666	5.55
18	0.9459	7.88	39	0.8284	6.90	60	0.7368	6.14	81	0.6635	5.52
19	0.9395	7.83	40	0.8235	6.86	61	0.7329	6.11	82	0.6604	5.50
20	0.9333	7.78	41	0.8187	6.82	62	0.7290	6.07	83	0.6573	5.48
21	0.9271	7.72	42	0.8139	6.78	63	0.7253	6.04	84	0.6542	5.45
22	0.9210	7.67	43	0.8092	6.74	64	0.7216	6.01	85	0.6511	5.42
23	0.9150	7.62	44	0.8045	6.70	65	0.7179	5.98	86	0.6481	5.40
24	0.9090	7.57	45	0.8000	6.66	66	0.7142	5.95	87	0.6451	5.38
25	0.9032	7.53	46	0.7954	6.63	67	0.7106	5.92	88	0.6422	5.36
26	0.8974	7.48	47	0.7909	6.59	68	0.7070	5.89	89	0.6392	5.33
27	0.8917	7.43	48	0.7865	6.55	69	0.7035	5.86	90	0.6363	5.30
28	0.8860	7.38	49	0.7821	6.52	70	0.7000	5.83	95	0.6222	5.18
29	0.8805	7.34	50	0.7777	6.48	71	0.6965	5.80	.	. .	. .
30	0.8750	7.29	51	0.7734	6.44	72	0.6930	5.78	.	. .	. .

TABLE IV.

Showing the Specific Gravity, Degrees Baumé, and Weight per Gallon and per Cubic Foot of Certain Oils.

	Specific Gravity.	Degrees Baumé.	Pounds in One Gallon.	Pounds in One Cubic Foot.
Water	1.0000	10	8.33	62.50
Castor Oil	.9639	15	8.03	60.24
Linseed Oil, boiled	.9411	19	7.84	58.81
Linseed Oil, raw	.9299	21	7.75	58.12
Menhaden, light	.9325	20	7.77	58.28
Menhaden, dark	.9292	21	7.74	58.08
Hemp-seed	.9307	20	7.75	58.17
Cod Liver	.9270	21	7.72	57.94
Whale	.9254	21	7.71	57.84
Poppy-seed	.9243	21	7.70	57.77
Cotton-seed	.9220	22	7.67	57.58
Fish	.9205	22	7 67	57.58
Olive	.9192	22	7.65	57.45
Almond	.9180	23	7.65	57.38
Lard	.9175	23	7.64	57.34
Rape-seed	.9155	23	7.63	57.22
Neat's-foot	.9142	23	7.62	57.14
Colza	.9136	23	7.61	57.10
Palm	.9046	25	7.54	56.54
Sperm, natural	.8815	29	7.34	55.09
Sperm, bleached	.8813	29	7.34	55.08
Spirits of Turpentine	.8600	33	7.16	53.75
Alcohol, 90 per cent.	.8228	40	6.85	51.43
Alcohol, 95 per cent.	.8089	43	6.74	50.56
Alcohol, absolute	.7938	46	6.61	49.61

NOTE.—In the column marked Baumé, the nearest whole number is given, omitting fractions.

TABLE V.

Comparison of Saybolt's A Viscosimeter and Doolittle's Viscosimeter.

Oil.	Specific Gravity.	Saybolt. Seconds.	Doolittle. Grams of Sugar.
Stove Gasolene	.680	23	0.0
Water	1.000	25	0.0
Kerosene, 150° Fire Test	.783	26.8	3.3
Lantern, 300° Fire Test	.827	36.4	49.8
Spindle	.849	57.9	62.8
Spindle	.851	60	65.5
Sperm	.880	102	73.5
Spindle	.863	115	74.3
Spindle	.879	138	77.0
Spindle	.868	156	78.4
Loom	.888	203	80.6
Lard	.916	215	82.8
Mineral	.907	224	83.5
Neat's-foot	.916	250	83.7
25° Paraffine	.900	272	84.2
Mineral	.903	339	86.0

NOTE.—Table V. was made from data obtained in the author's laboratory; Tables VI. and VII. are from those issued by George D. Feidt & Co., Philadelphia, makers of the torsion instrument.

The results of Tagliabue's instrument should agree very closely with those obtained by the use of Saybolt's.

TABLE VI.

A Comparison of Saybolt B and Torsion Viscosimeters, at 70° F.

Saybolt B. Seconds.	Torsion Grams Sugar per 100 C. C. Solution.	(Difference) Grams Sugar.	Saybolt B. Seconds.	Torsion Grams Sugar per 100 C. C. Solution.	(Difference) Grams Sugar.
$14\frac{2}{5}$	0.0		56	83.2	.2
15	9.2	9.2	57	83.4	.2
16	22.5	14.3	58	83.6	.2
17	36.5	14.0	59	83.8	.2
18	47.7	11.2	60	84.0	.2
19	53.5	6.8	61	84.2	.2
20	57.7	4.2	62	84.35	.15
21	60.6	2.9	63	84.5	.15
22	63.2	2.6	64	84.65	.15
23	65.6	2.4	65	84.8	.15
24	67.5	1.9	66	85.0	.2
25	69.2	1.7	67	85.15	.15
26	70.6	1.4	68	85.3	.15
27	71.6	1.0	69	85.45	.15
28	72.6	1.0	70	85.6	.15
29	73.4	.8	71	85.75	.15
30	74.0	.6	72	85.9	.15
31	74.6	.6	73	86.0	.1
32	75.2	.6	74	86.15	.15
33	75.6	.4	75	86.25	.1
34	76.0	.4	76	86.35	.1
35	76.5	.5	77	86.45	.1
36	76.9	.4	78	86.55	.1
37	77.4	.5	79	86.65	.1
38	77.8	.4	80	86.75	.1
39	78.2	.4	85	87.25	.50
40	78.6	.4	90	87.60	.35
41	79.0	.4	95	87.8	.20
42	79.3	.3	100	88.0	.2
43	79.7	.4	105	88.1	.1
44	80.1	.4	110	88.2	.1
45	80.4	.3	115	88.3	.1
46	80.75	.35	120	88.4	.1
47	81.0	.25	125	88.5	.1
48	81.3	.3	130	88.6	.1
49	81.6	.3	135	88.7	.1
50	81.85	.25	140	88.8	.1
51	82.2	.35	145	88.9	.1
52	82.4	.2	150	89.0	.1
53	82.6	.2	155	89.05	.05
54	82.8	.2	160	89.10	.05
55	83 0	.2			

NOTE.—Saybolt viscosimeter used gave a viscosity at 70° F. of $37\frac{3}{4}$ seconds for pure sperm oil of .886 specific gravity.

TABLE VII.

A Comparison of Saybolt C and Torsion Viscosimeters, at 212° F.

Saybolt C. Seconds.	Torsion Grams Sugar per 100 C. C. Solution.	(Difference) Grams Sugar.	Saybolt C. Seconds.	Torsion Grams Sugar per 100 C. C. Solution.	(Difference) Grams Sugar.
23	48.1		60	65.75	.2
24	49.2	1.1	61	65.95	.2
25	50.25	1.05	62	66.1	.15
26	51.3	1.05	63	66.25	.15
27	52.3	1.0	64	66.40	.15
28	53.2	.9	65	66.6	.20
29	54.0	.8	66	66.8	.2
30	54.75	.75	67	67.0	.2
31	55.5	.75	68	67.2	.2
32	56.2	.7	69	67.3	.1
33	56.85	.65	70	67.5	.2
34	57.5	.65	71	67.7	.2
35	58.0	.5	72	67.8	.1
36	58.5	.5	73	68.0	.2
37	59.0	.5	74	68.2	.2
38	59.45	.45	75	68.3	.1
39	59.85	.40	76	68.45	.15
40	60.25	.40	77	68.60	.15
41	60.6	.35	78	68.75	.15
42	60.9	.3	79	68.95	.2
43	61.25	.35	80	69.1	.15
44	61.6	.35	85	69.9	.8
45	61.95	.35	90	70.6	.7
46	62.3	.35	95	71.3	.7
47	62.6	.3	100	71.9	.6
48	62.9	.3	105	72.4	.5
49	63.2	.3	110	72.9	.5
50	63.45	.25	115	73.3	.4
51	63.7	.25	120	73.7	.4
52	64.0	.3	125	74.0	.3
53	64.3	.3	130	74.2	.2
54	64.5	.2	135	74.4	.2
55	64.7	.2	140	74.6	.2
56	64.9	.2	145	74.8	.2
57	65.15	.25	150	74.9	.1
58	65.35	.20	155	74.95	.05
59	65.55	.2	160	75.0	.05

NOTE.—Saybolt viscosimeter used gave a viscosity at 212° F. of 36½ seconds for pure sperm oil of .884 specific gravity.

TABLE VIII.

Showing the Principal Constants of Various Oils.

NAME.	Specific Gravity, 15° C.	Valenta, ° C.	Maumené, ° C.	Elaidin.	Iodine, Per cent.	Saponification, Mgrms. KOH.
Almond	.918	110	53	Solid.	97	190
Castor	.961	Soluble cold	47	....	84	178
Chinese Wood	.940	......	..	....	163	193
Cocoanut	.874[1]	......	..	....	8	260
Colza. (See Rape)						
Corn	.922	80	85	Pasty.	115	191
Cotton-seed	.922	90[2]–110	76	Pasty.	108	193
Elain	.904	......	..	....	90[3]	200
Horse	.919	54 68 80	52	....	80	197
Lard	.915	54 66 98	41	Very solid.	65	195
Linseed	.934	57[2]–74 70–79	111	....	176	191
Maize. (See Corn)						
Menhaden	.930	64[2]	126	....	154	190
Neat's-foot	.915	62 72–75	42	Solid at times.	68	194
Olive	.916	85–111	35	Very solid.	82	194
Palm	.859[1]	......	..	....	52	199
Peanut	.917	87[2]–112	51	Solid.	98	194
Poppy-seed	.925	......	87	....	138	193
Rape	.916	Insoluble.	55	Pasty.	101	174
Sesamé	.923	87[2]–107	65	Pasty.	107	190
Sperm	.880	Insoluble.	46	Solid.	83	135
Sunflower	.925	......	71	....	125	192
Tallow	.916	71–75	35	Solid.	56	197
Whale	.927	......	88	....	120	190

[1] At 99° C. [2] Allen. [3] Pure oleic acid.

TABLE IX.

Volumetric Factors.

1 Cc. $\frac{N}{2}$ HCl = .018185 g. HCl.

1 Cc. $\frac{N}{10}$ HCl = .003637 g. HCl.

1 Cc. $\frac{N}{2}$ KOH = .028 g. KOH.

1 Cc. $\frac{N}{6}$ KOH = .047 g. oleic acid = .008133 g. H_2SO_4.

1 Cc. $\frac{N}{10}$ KOH = .0056 g. KOH.

1 Cc. $K_2Cr_2O_7$ 3.8747 grms. per liter = .0038747 g. $K_2Cr_2O_7$ = .010 g. I.

1 Cc. $\frac{N}{10}$ $Na_2S_2O_3 + 5H_2O$ = .0248 g. $Na_2S_2O_3 + 5H_2O$ = .01265 g. I.

TABLE X.

The Action of Oils upon Metals.[1]

A. Oils.

Name.	No Action on	Least Action on	Most Action on
Cotton-seed	. . . .	Lead.	Tin.
Lard	. . . .	Zinc.	Copper.
Mineral	. . . . . Zinc.	Brass.	Lead.
Olive	. . . .	Tin.	Copper.
Rape	. . . . . . Brass and Tin.	Iron.	Copper.
Seal	. . . .	Brass.	Copper.
Sperm	. . . .	Brass.	Zinc.
Tallow	. . . .	Tin.	Copper.
Whale	. . . . . Tin.	Brass.	Lead.

B. Metals.

Brass	. . . . . . Rape.	Seal.	Olive.
Copper	. . . . . Mineral.	Sperm.	Tallow.
Iron	. . . .	Seal.	Tallow.
Lead	. . . .	Olive.	Whale.
Tin	. . . . Rape.	Olive.	Cotton-seed.
Zinc	. . . . Mineral.	Lard.	Sperm.

[1] I. J. Redwood, Jour. Soc. Chem. Ind., v. 362 (1886).

TABLE XI.

Showing the Principal Constants of Fatty Acids derived from Various Oils.

Name.	Melting Point.	Sap. Value.	Molec. Weight.	Iodine Value.
Almond	14° C.	204	. . .	95
Castor	13	. . .	290–295	87–88
Cocoanut	25–27	. . .	200	8–9
Corn	18–20	198.4	. . .	113–125
Cotton-seed	35–40	202–208	275–289	110–116
Horse	25–35	. . .	. . .	72–82
Linseed	22	197	283–307	159–185
Neat's-foot	17–26	. . .	. . .	64–69
Olive	21–28.5	198	280–286	86–90
Palm	47–50	206	270	. . .
Peanut	28–31	202	282	97
Poppy-seed	20	199	. . .	116–139
Rape	19	185	314	96–106
Sesamé	21–30	200	286	111
Sperm	11	. . .	. . .	83
Sunflower	17–24	201	. . .	124–134
Tallow	34–37	. . .	. . .	56
Whale	16	. . .	. . .	131

TABLE XII.

Showing the Bromine Addition and Substitution Figures. McIlhiney's Method (page 61).

	Total.	Addition.	Substitution.	Iodine.
Benzine[1]	51.5	15.5	18.0	. .
Benzine[1]	6.3	2.7	1.8	. .
Cocoanut Oil	5.4	4.7	0.3	. .
Cotton-seed	65.8	62.2	1.8	. .
Corn. (See Maize.)				
Linseed, average	112.0	106.6	2.7	183.8
Linseed, boiled, average	109.5	103.0	3.2	. .
Maize, average	75.8	72.9	1.5	. .
Menhaden, average	110.6	95.6	7.5	174.9
Paraffine, hard	3.5	1.4	1.1	. .
Petroleum, neutral	14.4	6.4	4.0	. .
Rosin, w. g.	161.4	8.0	76.7	. .
Rosin, black	135.4	5.4	65.0	. .
Rosin Oil	92.3	7.7	42.3	63.9
Rosin Oil, third run	197.6	16.4	90.6	. .
Tallow	24.0	21.5	1.3	. .
Turpentine	266.1	166.1	50.0	. .

[1] Substitutes for turpentine.

TABLE XIII.

Showing a Comparison of the Iodine Numbers obtained by Various Methods.

Oil.	Hübl.	Hanus.	Wijs.
Butter	35.3	35.3	36.2[2]
Castor	82.6	84.4	85.6[1]
Cod	148.5	147.5	154.6[1]
Cotton-seed	108.5	107.0	110.0[1]
Cocoanut	8.9	8.6	9.0[2]
Corn	119.0	120.2	123.2[2]
Lard	70.0	69.7	. . [2]
Linseed	179.5	183.7	188.7[2]
Oleomargarine	66.3	64.8	66.0[2]
Oleomargarine	52.5	52.0	52.9[2]
Oleomargarine	89.8	90.0	91.4[2]
Olive	79.2	80.6	79.9[2]
Peanut	96.3	97.4	99.0[2]
Poppy-seed	133.4	132.9	135.2[2]
Rape	100.2	102.8	104.1[2]
Sesamé	106.4	106.5	107.0[2]
Sunflower	106.4	107.2	109.2[2]
Whale	120.2	120.7	124.8[1]

[1] Hunt, Jour. Soc. Chem. Ind., xxi. 454.

[2] Tolman and Munson, Jour. Am. Chem. Soc., xxv. 244.

REAGENTS.

The reagents used in oil analysis are few and easily obtained. A list and their method of preparation is here given.

Acetic Acid, Glacial.—Kahlbaum's "Eisessig," ninety-nine and five-tenths per cent. pure. The determination of its strength should be made by titration and not by specific gravity, as the ninety-eight per cent. and eighty per cent. acid have the same specific gravity, 1.067. For Hanus's solution it must not reduce potassium bichromate and sulphuric acid.

Acetic Anhydride.—Kahlbaum's "Essigsäures Anhydrid."

Alcohol.—Commercial "Cologne Spirits." For the preparation of alcohol free from aldehyde for alcoholic potash, cologne spirits are treated with ordinary potassium hydrate, in the proportion of about twenty-five grams to the liter. The solution which forms is allowed to stand for a week to ten days and distilled, a few bits of pumice, prepared by igniting it and immediately quenching under water, being added to prevent bumping. Alcohol for use in the free acid determination is prepared by placing ten to fifteen grams of dry sodium carbonate in the reagent bottle, taking care to filter it before use.

Alcohol, Amyl.—Kahlbaum's manufacture.

Bromine.—The commercial article; also a $\frac{N}{3}$ solution, made by dissolving 26.6 grams bromine in one liter carbon tetrachloride.

Calcium Chloride.—The dry and also the crystallized salt.

Calcium Sulphate.—Plaster of Paris.

Carbon Tetrachloride.—Kahlbaum's "Tetrachlorkohlenstoff."

Chloroform.—Squibb's, U.S.P.

Copper.—Copper turnings or clippings, used for the generation of nitric oxide.

Copper Wire.—Cut in pieces of 0.3 to 0.5 gram.

Ether.—Squibb's, U.S.P.

Gasolene.—Gasolene, 86° Baumé.

Hydrochloric Acid, C.P.—Specific gravity 1.2. For $\frac{N}{2}$ HCl, dilute thirty-nine cubic centimeters of the above acid to one liter and standardize.

Iodine Solution.—Fifty grams of iodine to one liter of alcohol. For Hanus's solution dissolve by warming 13.2 grams iodine in one liter glacial acetic acid; cool and add three cubic centimeters of bromine.

Lead, Precipitated.—Place strips of zinc in the solution of lead acetate below. When the precipitation is nearly complete the lead is washed with water, alcohol, and ether, and dried finally in a vacuum desiccator.

Lead Acetate.—One hundred grams of the salt to one liter.

Litmus Paper.

Mercuric Chloride.—Sixty grams of the salt to one liter of alcohol.

Nitric Acid.—Specific gravity 1.84.

Phenolphthalein.—One gram of the substance to five hundred cubic centimeters of alcohol.

Meta-Phosphoric Acid.—A saturated solution of the commercial "stick phosphoric acid" in absolute alcohol.

Potassium Bichromate.—Dissolve 3.8747 grams of the C.P. salt in one liter of water; one cubic centimeter is equivalent to 0.01 gram of iodine. The solution should be tested against iron wire containing a known percentage of iron.

Potassium Hydrate.—$\frac{N}{2}$: Dissolve thirty grams of "potash by alcohol" in one liter of alcohol. $\frac{N}{4}$: Dissolve ten grams of ordinary "stick potash" in one liter of water and dilute to proper strength. The solution should be protected by stick potash from the carbon dioxide in the air. *Ten per cent.:* Dissolve one hundred grams of "stick potash" in eleven hundred cubic centimeters of alcohol.

Potassium Iodate.—A two per cent. solution.

Potassium Iodide.—One hundred grams of the commercial salt are dissolved in one liter of water. This should be free from iodate, shown by yielding no coloration when acidified with strong HCl.

Silver Nitrate.—Thirty grams to one liter + 0.4 Cc. HNO^3.

Sodium.

Sodium Chloride.—Ordinary "coarse fine" salt for freezing mixtures.

Sodium Hydrate.—36° Baumé. Dissolve three hundred grams of caustic soda in one liter of water.

Sodium Nitroprusside.—The commercial salt.

Sodium Thiosulphate.—$\frac{N}{10}$: Dissolve twenty-six grams of "sodium hyposulphite" in one liter of water; the addition of two grams of ammonium carbonate to the liter is said by Mohr to improve the stability of the solution.

Starch Solution.—Rub up in a mortar one gram of potato starch with ten to fifteen cubic centimeters of water, pour this into two hundred cubic centimeters of water which are boiling actively, and continue the boiling for a few minutes.

Sugar.—Ordinary granulated sugar.

Sulphur.—A 1.5 per cent. solution in carbon bisulphide.

Sulphuric Acid, C.P.—This should be at least ninety-nine and five-tenths per cent. pure, and its strength be determined by titration, as one hundred per cent. and ninety-four and three-tenths per cent. acid have the same specific gravity, 1.8384.[1]

Dilute.—One part acid to ten parts of water.

Nitrosulphuric Acid, for the Elaidin Test.—A liter of sulphuric acid of 46° Baumé (1.47 specific gravity) is prepared by diluting five hundred and sixty cubic centimeters commercial sulphuric acid to one liter; a few drops of nitric acid are added and nitric oxide (generated from copper and nitric acid) passed in until it is satu-

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[1] Richmond, Jour. Soc. Chem. Ind., ix. 479 (1890).

rated. The acid is then cooled in ice-water and the gas passed in until it is saturated at 0° C. This is called Roth's liquid.

Tin Tetrabromide.—This is prepared[1] by allowing bromine to fall drop by drop upon granulated tin contained in a dry flask immersed in cold water until the coloration shows bromine to be in excess. A small quantity of bromine is then added and the liquid diluted with three to four times its volume of carbon bisulphide.

OILS FOR RAILROAD USE.

The railroads being among the largest users of oil, their requirements are of interest; as they do not differ widely, those of the Philadelphia and Reading Railroad will serve as a sample.

Specifications for Lard Oil.

When a shipment of oil is received a sample will be taken at random from each sixty barrels or fraction thereof, and forwarded to the Test Department. This sample will be examined and the entire shipment accepted or rejected on its merits. If rejected the shipment will be returned at the shipper's expense.

Two grades of Lard Oil will be used, "Prime" and "Extra No. 1;" the former for burning purposes chiefly, and the latter as a lubricant. The material desired under this specification is oil from fresh lard of corn-fed hogs, unmixed with other oils. It should contain the least possible amount of free acid, and from October 1 to May 1 show a cold test not higher than 40° F.

Prime Lard Oil.

This grade of oil must not contain admixtures of any other oils or more free acid than is neutralized by four cubic centimeters of alkali, as described below.

[1] Allen, Commercial Organic Analysis, ii. 463.

Between October 1 and May 1 it must show a cold test below 45° F.

When tested with Nitrate of Silver, as described below, it must not show any coloration.

Extra No. 1 Lard Oil.

This grade of oil must not contain admixtures of any other oils or more free acid than is neutralized by thirty cubic centimeters of alkali, as described below.

Between October 1 and May 1 it must show a cold test below 45° F.

The Cold Test.—The cold test is made as follows:

About two ounces of oil is put in a four-ounce sample bottle, a thermometer inserted, and the oil frozen with ice, salt being used if necessary. When the oil is hard, the bottle is taken from the freezing mixture and the frozen oil stirred thoroughly with the thermometer until it will flow. The reading of the thermometer is then taken, and this temperature is regarded as the cold test of the oil.

Free Acid Test.—The solutions required for this test are ninety-five per cent. alcohol neutralized with sodium carbonate, caustic potash solution of such a strength that 31.5 cubic centimeters of it will exactly neutralize five cubic centimeters of a normal solution of sulphuric acid (forty-nine grams per liter), and a small amount of Phenolphthalein dissolved in Alcohol, and rendered neutral with caustic potash, to be used as an indicator.

Now weigh or measure into a four-ounce sample bottle 8.9 grams of the oil to be tested, add about two ounces of Alcohol, warm to about 150° F., and add a few drops of the Phenolphthalein.

Then run in the caustic potash from a graduated burette, with frequent shaking, until a permanent pink color remains after vigorous shaking. When this point is reached read the number of cubic centimeters used.

Nitrate of Silver Test.—Solution of Nitrate of Silver is made as follows:

Nitrate of Silver, 1 gram; Alcohol, 200 grams; Ether, 40 grams. After the ingredients are dissolved and mixed, allow the solution to stand in a bright light until it has become perfectly clear; it is then ready for use, and should be kept in a dim place, and tightly corked.

Into a fifty cubic centimeter test-tube put ten cubic centimeters of the oil to be tested, previously filtered through washed filter-paper. Add five cubic centimeters of the above solution, shake thoroughly, and heat in a vessel of boiling water fifteen minutes with occasional shaking. If the oil is satisfactory it will show no change of color under this test.

Specifications for Petroleum Products.

When a shipment of oil is received, a sample shall be taken at random and forwarded to the Test Department. This sample will be examined and the entire shipment accepted or rejected on its merits. If rejected, the shipment will be returned at the shipper's expense.

150° Fire Test Oil.

This grade of oil shall be water-white in color, showing a flashing point not below 130° F., and a burning point not below 151.° The test will be made in an open vessel by heating the oil not less than ten degrees per minute, and applying the test flame every seven degrees, beginning at 123°. The gravity may be from 46° to 50° Baumé. Oil will not be received which is cloudy from the presence of glue or suspended matter of any kind.

300° Fire Test Oil.

This grade of oil shall be water-white in color, show a flashing point not below 256° F., and a burning point not below 298°. The

test will be made in an open vessel by heating the oil not less than fifteen degrees per minute, and applying the test flame every seven degrees, beginning at 249°.

When heated to a temperature of 425° and held there for five minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter,—either at this temperature or on cooling.

When the oil is cooled to the temperature of 32°, and held there for ten minutes, it must remain clear and transparent, showing no cloudiness. The gravity may be from 38° to 42° Baumé.

Oil will not be received which is cloudy from the presence of glue or suspended matter of any kind.

Car Oil.

This grade of oil, commonly known as Well Oil or Black Oil, should have a gravity of about 29° Baumé, and must not show a flashing point below 325° F. The test will be made in an open vessel by heating the oil not less than fifteen degrees per minute, and applying the test flame once in seven degrees, beginning at 304°.

Oil received during the months of August and September must have a cold test not above 15° F., and from October 1 to April 1, a cold test not above 5° F. when determined as described below.

From August 1 to April 1, at 80° F., the oil must show a viscosity not lower than that of a pure cane sugar solution containing eighty grams of sugar in one hundred cubic centimeters of the syrup, and at 150° F. a viscosity not lower than that of a pure cane sugar solution containing sixty-six grams of sugar in one hundred cubic centimeters of the syrup, the viscosity of the sugar solution being taken at 80° F.

From April 1 to August 1, at 80° F., the oil must show a viscosity not lower than that of a pure cane sugar solution containing eighty-eight grams of sugar in one hundred cubic centimeters of

the syrup, and at 150° F. a viscosity not lower than that of a pure cane sugar solution containing sixty-eight grams of sugar in one hundred cubic centimeters of the syrup, nor higher than that given by a pure cane sugar solution containing seventy-five grams of sugar in one hundred cubic centimeters of the syrup, the viscosity of the sugar solutions being taken at 80° F.

The oil must be transparent, with a reddish-brown or greenish color, free from lumps or specks.

No oil will be accepted which shows more than five per cent. of flocculent or tarry matter settled out after five cubic centimeters of the oil have been mixed with ninety-five cubic centimeters of 88° Gasolene, and allowed to stand for an hour.

CYLINDER STOCK.

This grade of oil shall show a flashing point not below 525° F., and a burning point not below 600° F. The test will be made in an open vessel by heating the oil not less than twenty degrees per minute, and applying the test flame every seven degrees, beginning at 504°.

This oil must flow readily at 60° F., and at 350° F. must show a viscosity not lower than that of a pure cane sugar solution containing fifty-eight grams of sugar in one hundred cubic centimeters of the syrup, the viscosity of the sugar solution being taken at 80° F.

The oil must be transparent, with a reddish-brown or greenish color, free from lumps or specks.

No oil will be accepted which shows more than five per cent. of flocculent or tarry matter settled out after five cubic centimeters of the oil have been mixed with ninety-five cubic centimeters of 88° Gasolene, and allowed to stand for one hour.

Cold Test.—About two ounces of oil are put in a four-ounce sample bottle, a thermometer inserted, and the oil frozen with a mixture of ice and salt. When the oil is hard the bottle is taken

from the freezing mixture and the frozen oil stirred thoroughly with the thermometer until it will flow. The reading of the thermometer is then taken, and this temperature is regarded as the cold test of the oil.

NOTE.—The viscosity tests will be made upon the Torsion Viscosimeter.

Manufacturers not having this instrument may submit a sample of oil to the Test Department, and will be furnished with the information necessary to standardize the viscosimeter they may have in use.

Specifications for Compound Oils.

When a shipment of oil is received, a sample shall be taken at random and forwarded to the Test Department. This sample will be examined and the entire shipment accepted or rejected on its merits. If rejected, the shipment will be returned at the shipper's expense.

CYLINDER OIL.

This oil shall consist of a high grade cylinder stock, compounded with not less than twenty per cent. by weight of acidless animal oil, Tallow or Tallow Oil being preferred.

The compounded oil shall show a flashing point not below 525° F., and a burning point not below 600°. The test will be made in an open vessel by heating the oil not less than twenty degrees per minute, and applying the test flame every seven degrees, beginning at 504°.

This oil must flow readily at 60° F., and at a temperature of 350° F. must show a viscosity not lower than that of a pure cane sugar solution containing fifty-eight grams of sugar in one hundred cubic centimeters of the syrup, the viscosity of the sugar solution being taken at 80° F.

The oil must be transparent, with a reddish-brown or greenish color, free from lumps or specks.

No oil will be accepted which shows more than five per cent. of flocculent or tarry matter settled out after five cubic centimeters of the oil have been mixed with ninety-five cubic centimeters of 88° Gasolene, and allöwed to stand for one hour.

Signal Oil.

This grade of oil shall be prime white in color, shall contain not less than forty per cent. by weight of Prime Lard Oil, and shall show a flashing point not below 200° F., and a burning point not above 300°. The test will be made in an open vessel by heating the oil not less than fifteen degrees per minute, and applying the test flame every seven degrees, beginning at 193°.

When heated to a temperature of 450°, and held there for five minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter, either at this temperature or on cooling. The gravity may be from 31° to 34° Baumé.

Oil will not be received which is cloudy from the presence of glue or suspended matter of any kind.

No. 1 Engine Oil.

This oil shall consist of a high grade of mineral oil, compounded with not less than ten per cent. by weight of nearly acidless animal oil.

It shall show a gravity of about 29° Baumé, and a flashing point not below 325° F. The test will be made in an open vessel by heating the oil not less than fifteen degrees per minute, and applying the test flame once in seven degrees, beginning at 304°.

Oil received during the months of August and September must have a cold test not above 15° F., and from October 1 to April 1 a cold test not above 5° F., when determined as described below.

From August 1 to April 1, at 80° F., the oil must show a viscosity not lower than that of a pure cane sugar solution containing

eighty grams of sugar in one hundred cubic centimeters of the syrup, and at 150° a viscosity not lower than that of a pure cane sugar solution containing sixty-six grams of sugar in one hundred cubic centimeters of the syrup, the viscosity of the sugar solution being taken at 80° F.

From April 1 to August 1, at 80° F., the oil must show a viscosity not lower than that of a pure cane sugar solution containing eighty-eight grams of sugar in one hundred cubic centimeters of the syrup, and at 150° F. a viscosity not lower than that of a pure cane sugar solution containing sixty-eight grams of sugar in one hundred cubic centimeters of the syrup, nor higher than that given by a solution of pure cane sugar containing seventy-five grams of sugar in one hundred cubic centimeters of the syrup, the viscosity of the sugar solutions being taken at 80° F.

The oil must be transparent, with a reddish-brown or greenish color, free from lumps or specks.

No oil will be accepted which shows more than five per cent of flocculent or tarry residue settled out after five cubic centimeters of the oil have been mixed with ninety-five cubic centimeters of 88° Gasolene, and allowed to stand for an hour.

No. 2 Engine Oil.

The requirements for this oil are identically the same as those for No. 1 Engine Oil, with the following exceptions:

It must contain not less than twenty per cent. by weight of nearly acidless animal oil.

From October 1 to April 1 the cold test must be not above 10° F. when determined as described below.

Screw-Cutting Oil.

This oil shall consist of paraffine oil of about 27° Baumé gravity, compounded with not less than twenty-five per cent. by weight of Fat Oil, Cotton seed preferred.

The compound oil shall show a flashing point not below 300° F., and a burning point not above 425°. The test will be made in an open vessel by heating the oil not less than fifteen degrees per minute, and applying the test flame once in seven degrees, beginning at 276°.

From October 1 to April 1 the oil must have a cold test not above 15° F. when determined as described below.

Cold Test.—About two ounces of oil are placed in a four-ounce sample bottle, a thermometer inserted, and the oil frozen with a mixture of ice and salt. When the oil is hard, the bottle is taken from the freezing mixture, and the frozen oil stirred thoroughly with the thermometer until it will flow. The reading of the thermometer is then taken, and this temperature is regarded as the cold test of the oil.

NOTE.—The viscosity tests will be made upon the Torsion Viscosimeter.

Manufacturers not having this instrument may submit a sample of oil to the Test Department, and will be furnished with the information necessary to standardize the instrument they may have in use.

Specifications for Tallow.

Tallow to be used for cylinder lubrication should be rendered as soon as possible after the animal is killed, in order to have the amount of free acid as small as possible.

Tallow which on examination is found to contain dirt or cracklings disseminated through it, or which has a layer of dirt or cracklings in the bottom of the barrel more than an eighth of an inch thick, will be rejected.

Tallow will not be accepted which has more free acid than can be neutralized by three cubic centimeters of the alkali solution used for this determination (p. 148), or which contains any foreign substance not properly belonging to tallow.

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THE END.